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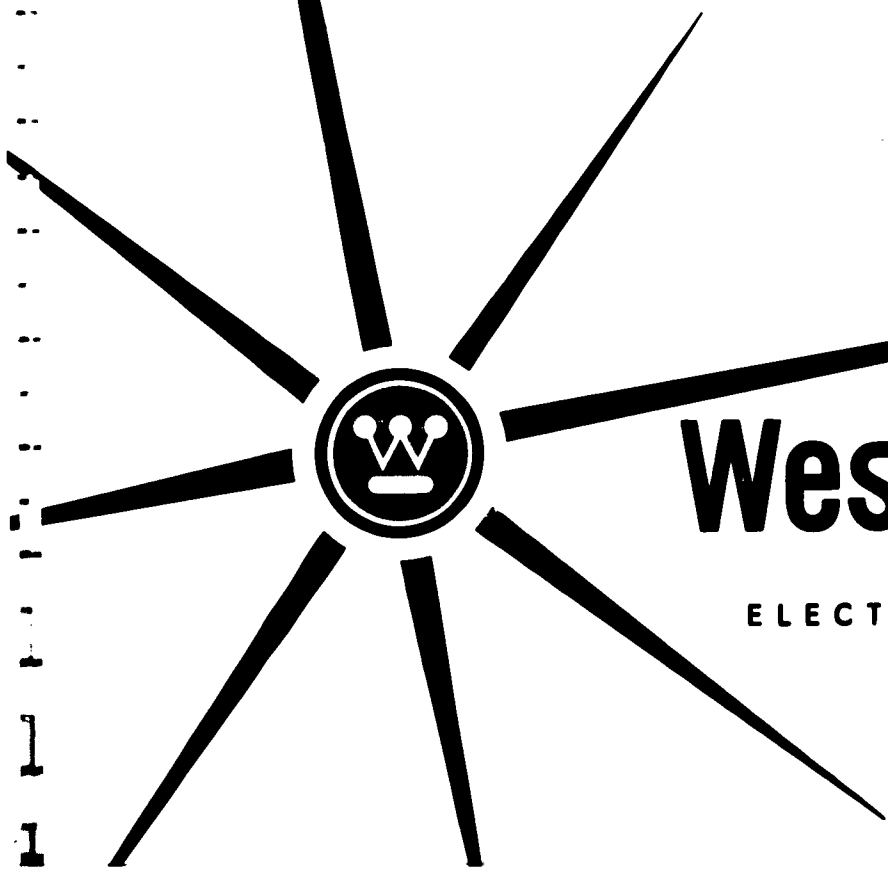
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**Westinghouse**

ELECTRIC CORPORATION

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November 15, 1963

This Quarterly Report covers the period August 15, 1963, to  
November 15, 1963, on Contract AF33(657)10701, Task No. 8128-08.

Prepared by:

E. S. BOBER W.H.S.  
E. S. Bober, Supv'y Engr.

W. H. Snively  
W. H. Snively, Adv. Scientist

R. E. Stapleton  
R. E. Stapleton, Proj. Engr.

D. K. McIlvaine  
Supervised by D. K. McIlvaine,  
Proj. Mgr.

J. D. Miner  
Approved by J. D. Miner,  
Manager of Engineering

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### NOTICE

The work covered by this report was accomplished under Air Force Contract AF33(657)10701, but this report is being published and distributed prior to Air Force review. The publication of this report, therefore, does not constitute approval by the Air Force of the findings and conclusions contained herein. It is published for the exchange and stimulation of ideas.

**DEVELOPMENT OF HIGH TEMPERATURE  
ALKALI METAL RESISTANT INSULATED WIRE**

**3rd Quarterly Progress Report  
Contract AF33(657)10701, Task No. 8128-08**

**November 15, 1963**

**Westinghouse Electric Corporation  
Aerospace Electrical Division  
Lima, Ohio**

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## FOREWORD

This report is submitted by the Aerospace Electrical Division, Westinghouse Electric Corporation, Lima, Ohio, on Air Force Contract AF33(657)10701, Task No. 8128-08, "Development of High Temperature Alkali Metal Resistant Insulated Wire". The contract is administered by the Aeronautical Systems Division, Wright Patterson Air Force Base, Dayton, Ohio. Mr. Lester Schott is project engineer.

The work described in this report was carried out by personnel at the Research and Development Center, Department of Insulation and Chemical Technology, Westinghouse Electric Corporation, Pittsburgh, Pennsylvania.

## ABSTRACT

This report covers the progress during the third quarter on Air Force Contract AF33(657)10701. The program effort is directed toward the development of an insulated electrical conductor resistant to saturated potassium (850 C) and mercury (538 C) vapors. Fifty-two potassium and twenty-one mercury corrosion capsules containing test specimens were prepared during this quarter. Conductivity measurements were conducted at high temperature (538 C) and (850 C) on clad conductors. Several attempts to fabricate electrical test capsules for mercury and potassium exposure were partially successful.

Nickel clad silver coils of a No. 8 wire size have been successfully coated with several different ceramic coatings by plasma spraying. The coated wire coil could be compressed without destroying the ceramic insulation. A series of active metal brazing alloys were tested for their corrosion resistance to potassium vapors at 850 C. Several alloys were found to be resistant to this environment. Aluminum nitride coatings, formed by decomposition of an aluminum chloride-ammonia complex, were investigated as a possible ceramic insulation resistant to potassium vapor at 850 C.

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## I. INTRODUCTION

Rotary power sources for advanced weapon systems based on nuclear reactors as energy sources utilize liquid metals as the working fluid to drive the turbines. Since the a-c generator used to supply the electrical energy is attached to the turbine shaft, its electrical insulation would be exposed to any of the metal vapor leaking through the seals. Present electrical insulation will probably be severely attacked when exposed to high temperature mercury vapor or alkali metal vapor such as potassium. In order to provide reliable electrical power, the present insulation must be protected from metal vapor by stator canning techniques.

Under this contract, a program was initiated to investigate insulation materials, electrical conductors, and coating methods needed in the development of a high current (4000 amps per square inch) round wire for advanced electromagnetic alternators that are exposed to mercury and alkali metal vapors. Saturated mercury vapor at 538 C was chosen to provide a realistic vapor pressure in test cells. Saturated potassium vapor at 850 C was chosen as a representative alkali metal vapor condition. The design objective life of the insulated conductor in the metal vapor environment is 10,000 hours. The electrical resistance of the conductor at test temperature during its life is not to exceed 150% of the copper standard at 850 C. The room temperature tensile strength of the conductor is to be in excess of 30,000 psi. The initial purity of the potassium used in the

exposure tests is 99.97%.

It is preferred that the conductor be insulated with a compatible high temperature insulation that is resistant to metal vapor attack, however, if this is not possible, then a potting compound compatible with the insulation and resistant to the metal vapor will be evaluated. The electrical strength from conductor to ground should have a design objective of 1200 volts. If the insulated conductor is potted, the electric strength of the insulation should be at least 300 volts per mil.

The final evaluation of the insulated wire will be done in stator-ettes. The insulated conductor coils will be inserted in test stator-ettes to investigate the effect of test environments. While in the statorette, the insulation system will be subject to metal vapors, temperature, thermal shock, nuclear radiation, vibration, mechanical shock, humidity and acceleration.



## II. SUMMARY OF WORK PERFORMED AND MAJOR RESULTS

1. Fifty-two potassium and twenty-one mercury corrosion capsules containing test specimens were prepared during this quarter. Five additional test capsules incorporating commercial, lead-wire insulator assemblies were also prepared for initial electrical tests in a mercury vapor environment.
2. Tantalum tubes closed by means of stainless steel Swagelok units developed leaks at temperatures of 400-500 C. One Swagelok unit machined from tantalum has been received for trial use in sealing tantalum capsules.
3. A three hundred foot length of AWG No. 8 nickel clad silver wire was received from Sylvania. Delivery of a six hundred foot length of AWG No. 8 Inconel clad silver wire from Sylvania originally scheduled for early October has been rescheduled for late December.
4. Samples of commercial grade copper wire were aged in argon at 540 C and 850 C to check the equipment and techniques used to measure electrical conductivity of conductors. The values measured at 540 C and 850 C on the specimens were within 2% and 3% respectively of the values calculated for the wire.

5. The average conductivity measured on chrome plated and iron plated OFHC copper wire aged in argon at 540 C for 500 hours was 97% and 87% respectively of that calculated for copper at 540 C. The aging resulted in a permanent decrease in the conductivity of the iron plated OFHC copper wire.
6. Two specimens of Oxalloy 28 wire were aged in argon at 850 C for 500 hours. Conductivity of the two samples dropped below the minimum value (67% of Cu standard) after 400 hours and 100 hours respectively.
7. Pre-exposure of Oxalloy 28 to mercury vapor at 540 C for 360 hours did not result in any significant change in conductivity of the wire during 500 hours aging at 540 C in argon.
8. The electrical conductivity of Sylvania's nickel clad (20%) silver wire remained a constant 89% of the calculated value for copper during a 500 hours aging at 850 C in argon.
9. The volume resistivity at 540 C was determined for seven insulators, four of which had been pre-exposed to mercury vapor at 540 C for 260 hours. Beryllia, the only material in which a direct comparison of vapor exposure effects was possible, decreased from  $10^{10}$  to  $10^4$  ohm-inches. The resistivity of strontium zirconate after vapor exposure was  $1.4 \times 10^7$  ohm-inches at 540 C.

10. The volume resistivity at 850 C was determined for six insulators, three of which had been pre-exposed to potassium vapor at 850 C for 165 hours. Hot pressed magnesia, the only material on which a direct comparison of vapor effects was possible, decreased from  $2.5 \times 10^6$  to  $7.8 \times 10^3$  ohm-inches. The resistivity of strontium zirconate after vapor exposure was  $6.5 \times 10^4$  ohm-inches at 850 C.
11. Electrical leakage resistance in argon at 850 C measured on an experimental lead in assembly was  $9.7 \times 10^7$  ohms. The assembly consisted of a 1/2" dia. Lucalox disk brazed with 95% Zr-5%Be into the ID of a tantalum tube and having a molybdenum lead wire sealed in a hole through the Lucalox disk with the same alloy.
12. Electrical volume resistivity and leakage resistance at 850 C in argon were determined for a stainless steel clad, magnesia powder insulated OFHC copper conductor supplied by Advanced Technology Laboratory. The measured values were  $1.4 \times 10^8$  ohms per inch respectively.
13. Electric leakage resistance measurements were made on the five mercury vapor filled capsules incorporating the commercial lead in assemblies. All units developed leaks prior to attaining a temperature of 540 C (at insulator). A typical resistance value measured before leakage occurred was  $4 \times 10^7$  ohms (insu-

lator at 400 C - mercury reservoir at 380 C).

14. Lucalox, sapphire and metals including tantalum were successfully joined using a 95%Zr-5%Be brazing alloy. The alloy showed some signs of attack after 170 hours exposure to potassium vapor at 850 C. However, the attack was mild enough to consider use of the alloy in short term (100 hours) exposures to potassium vapor.
15. Three tantalum tube capsules using the 95%Zr-5%Be brazing alloy were prepared for use in electrical tests in potassium vapor. One capsule had a molybdenum lead wire through a Lucalox insulator, the second a tantalum lead wire through a sapphire insulator and the third a tantalum lead wire through a Lucalox insulator. The interior metal surfaces of the third capsule were heavily plated with nickel.
16. A 68% Ti-28% V-4%Be brazing alloy exhibited fair resistance to potassium vapor at 850 C during a 170 hour exposure; however, attempts to use it to seal Lucalox disks to the ID of tantalum tubes results in severe cracking of the Lucalox.
17. Joints of tantalum-to-Kovar-to-stainless-steel have successfully been prepared for exposure to potassium vapor at 850 C. If they are found resistant, the capsules in 15 above will be modified in this way to permit sealing by means of stainless

steel Swageloks.

18. Various oxides have been successfully applied by plasma spray techniques to Inconel, nickel, and stainless steel substrates. Preformed coils of the nickel clad silver wire have been satisfactorily coated with alumina.
19. A silica free fusion coating, that will fuse at or slightly above the maximum safe conductor temperature, has not been developed to date. Brief exposure of selected coatings to temperatures of 1400 C has produced no significant results.
20. Powdered alumina in colloidal zirconia produced adherent, uniform coatings on stainless steel, Inconel and nickel substrates when fired at 1000 C.
21. Decomposition of an  $\text{AlCl}_3 \cdot \text{NH}_3$  complex on graphite surfaces at 1000 C produced well bonded, continuous insulating films. X-ray diffraction patterns obtained on the films correspond to the hexagonal wurtzite crystal structure which AlN is known to have. Coatings were also formed on Mo and W wires. Insulating coatings could be formed on Oxalloy 28 wire only after it was well precoated with plasma sprayed alumina.
22. The BN coating formed by thermal decomposition of trichloroborazole was found to provide some protection to surfaces

exposed to potassium vapor at 850 C for 172 hours. The coating on wire specimens powdered somewhat during the metal vapor exposure, but the film remaining was nonconducting. Infrared spectra data on the coatings indicate them to be BN, but of varying crystallinity. Only films formed at temperatures of 1400 C or higher had the desired hexagonal structure. Also, the coatings tend to hydrolize and give off an ammonia odor on standing at room temperature in air.

### III. EXPERIMENTAL WORK AND EVALUATION OF RESULTS

#### 3.1 Metal Vapor Exposure - Mercury and Potassium

##### 3.1.1. Test Capsules - Design, Loading, and Sealing

The test capsule design used for the corrosion tests in the third quarter, is the same as that used during the previous quarter. Loading and sealing techniques also remained the same except for two modifications which were incorporated in the capsule sealing technique.

First, the electric impact wrench which formerly was used to seal the evacuated capsules was discarded in favor of a vertical shaft passing through the top of the dry box and sealed to it through a four unit rubber O-ring seal. By equipping this shaft with the appropriate socket and bar arrangement, Swageloks can now be sealed manually. It was felt that the modification was necessary because of frequent failures experienced with the electric-wrench and excessive outgassing during operation due to armature and stator winding deterioration.

A new grip and socket for sealing one inch outside diameter tubes with Swageloks was also built. With this addition, it is now possible to seal under evacuated conditions specimens

up to 13/16" in diameter.

Attempts to obtain a tantalum tube to stainless Swagelok joint on a loaded capsule, that remained vacuum tight up to 540 C, were unsuccessful. A stainless liner was pressed into the tantalum tube before sealing and increased this upper temperature capability by about 100 C. Since a number of promising metal to ceramic seals use tantalum, a Swagelok fabricated from this refractory metal has been obtained. It is believed that the tantalum to tantalum seal will remain tight over the necessary temperature range, and be capable of re-use by coating the Swagelok threads with a high temperature release agent before sealing.

An Inconel retort has been designed and purchased for use as a liner in an existing Lindbergh furnace for controlled atmosphere tests. The retort is equipped with a water cooled O-ring sealed face plate and contains the necessary plumbing to permit regulation of inert gas flow through the liner. Measurement of temperature and electrical parameters are accomplished by electrical feed-through terminals. The liner is capable of operation at 850 to 900 C and is currently being installed in the furnace.

### 3.1.2. Exposure Tests

During the past quarter, a total of fifty-two potassium and



twenty-four mercury corrosion capsules were prepared. Of these, five potassium and six mercury exposures still remain on test. The results of these tests are summarized in Tables 1 and 2. The majority of samples tested during this period were either brazing alloys of various compositions or ceramic to metal seals utilizing high temperature brazing alloys.

### 3.1.3. Evaluation of Corrosion Effects

For the sake of continuity, the discussion of the effects of the vapor exposure on specific materials has been included in the section of the report concerned with that type of material.

It can be seen from Table 1 that data in ten potassium vapor tests were lost due to heavy corrosion of the capsules. Figures 1 and 2 show one set of such capsules on removal from the aging oven. It will be noted in Figure 2 that the bottom half of the capsules are severely attacked. It is theorized that one of the capsules leaked due to a defective weld or Swagelok fitting, and the leaking potassium vapor oxidized and settled into the 1" OD receptacles holding the remaining capsules. The higher corrosive oxide or hydroxide then attacked the type 304 stainless steel resulting in eventual punctures of all tubes mounted in this way. The fact that two capsules standing free along side the holding block suffered no attack and remained tight supports this theory.

## 3.2 Electrical Tests

### 3.2.1. Testing in Argon

#### 3.2.1.1. Conductors

For mercury vapor applications, chrome-plated copper and iron-plated copper wire samples were thermally aged at 538 C for 500 hours in atmospheres of argon. The surface plate on each of these 12-inch long 50 mil diameter (No. 16 AWG) copper samples was about 2 mils. Electrical conductivity measurements were made on each conductor sample during its thermal aging period. A No. 16 gauge commercial grade copper wire sample was also thermally aged at 538 C for 500 hours in argon to provide a check on our ability to measure electrical conductivity. None of the above mentioned sample conductors had previous aging in mercury vapor. The measured conductivity of the commercial copper wire is plotted on Figures 3 and 4. A 5-inch length of "Oxalloy 28" wire (No. 16 gauge) previously exposed to 538 C mercury vapors for 360 hours was additionally aged, at the same temperature for 500 hours, in argon. Electrical conductivity data were secured for this conductor as it thermally aged in argon.

Results of electrical conductivity measurements at 538 C are shown in Table 3. The conductivity value shown for each

conductor represents an average of about 15 measurements made during its thermal aging time. The respective electrical conductivities of each conductor remained quite constant throughout their 500 hour thermal aging periods. Previously determined electrical conductivity of "Oxalloy 28" at 538 C is also included in Table 3 for purposes of comparison.

It is noted that the measured electrical conductivity of copper at 538 C and the calculated value agree within 2%. The close agreement with actual measurements justifies continued usage of the calculated electrical conductivity of copper as a standard to compare with measured values of other conductors.

The electrical conductivities of all samples shown in Table 3 are well above the lower allowable limit (67% of copper). Average conductivity of the pre-mercury exposed "Oxalloy 28" conductor is nearly identical in value to that found for unexposed "Oxalloy 28". It is concluded that the electrical conductivity of "Oxalloy 28" is unaffected by exposure to mercury vapors at a temperature of 538 C for a period of 360 hours.

Pre and post thermal aging electrical conductivity-temperature data were secured to 538 C for all conductors. With one exception, good agreement was found, indicating that the electrical conductivity was not adversely affected by thermal

aging in 538 C argon for 500 hours. The surfaces of all sample conductors showed degrees of darkening, however, this apparently had little effect on their measured electrical conductivities.

#### 3.2.1.2. Conductors - For Potassium Vapor Application

Three 12-inch long wire conductors were thermally aged at 850 C in purified argon. One conductor is 20% nickel clad-silver wire and is 128 mils in diameter (No. 8 AWG). The second conductor is "Oxalloy 28", 28% stainless steel clad-copper, and is 51 mils in diameter (No. 16 AWG). The electrical conductivity data obtained during the Oxalloy 28 re-run is shown in Figures 5 and 6. A No. 16 gauge commercial grade copper wire was also thermally aged to provide a means for checking our ability to measure electrical conductivity at 850 C. The electrical conductivity data obtained is shown in Figures 7 and 8. None of these conductors had previous aging in potassium vapor.

Results of electrical conductivity measurements at 850 C are shown in Table 4. The conductivity value shown for each conductor represents an average of about 15 measurements during its thermal aging time.

The measured electrical conductivity of copper at 850 C and

the calculated value agree within 3%. This close agreement with actual measurements justifies continued usage of the calculated electrical conductivity of copper as a standard to compare with measured values of other conductors.

Table 4 reveals that the conductivity of "Oxalloy 28" decreased with time and fell below the lower allowable limit, 67% of the electrical conductivity of copper, after 400 hours of aging at 850 C. A second test of a similar sample of "Oxalloy 28" failed after 100 hours of thermal aging. (Figures 5 and 6)

Electrical conductivity of the nickel clad-silver wire at 850 C remained constant throughout the 500 hour aging period. The average measured conductivity of this conductor,  $1.1 \times 10^5$  mhos per centimeter, is 89% of the calculated value for pure copper at 850 C. Thermal aging had very little effect on this conductor as the pre and post conductivity-temperature data agree quite well.

#### 3.2.1.3. Insulators for Mercury Vapor Application

The electrical volume resistivities of seven materials were determined in an atmosphere of tank argon at 538 C ambient temperature. The materials and their respective electrical resistivities are shown in Table 5. Four of the material samples had been previously exposed to 538 C mercury vapors

for 260 hours prior to the electrical measurements in argon. However, only one material, beryllia, allows direct comparison of the effect exposure to 538 C saturated mercury vapor has on its electrical resistivity. Pre-exposing beryllia to mercury vapors resulted in a drastic reduction in its volume resistivity from  $10^{10}$  to  $10^4$  ohm-inches at 538 C. Of the four materials previously exposed to saturated mercury vapors, strontium zirconate (Y-628) has the highest measured electrical volume resistivity,  $1.4 \times 10^7$  ohm-inches. This value is comparable to the "as received" hot pressed magnesia sample ( $1.9 \times 10^7$  ohm-inches).

#### 3.2.1.4. Insulators for Potassium Vapor Application

The electrical volume resistivities of six materials were determined in an atmosphere of tank argon at 850 C ambient temperature. These materials and their respective electrical resistivities are shown in Table 6. Three of the samples had been previously exposed to 850 C potassium vapor for 165 hours prior to the electrical measurements in argon. However, only one material, hot pressed magnesia, allows direct comparison of the effect exposure to 850 C saturated potassium vapor has on its electrical resistivity. Pre-exposing hot pressed magnesia to potassium vapors resulted in a reduction of its volume resistivity from  $2.5 \times 10^6$  to  $7.8 \times 10^3$  ohm-inches at 850 C. Of the three materials previously exposed to saturated

potassium vapors strontium zirconate (Y-628) has the highest measured electrical volume resistivity,  $6.5 \times 10^4$  ohm-inches. Strontium zirconate (Y-628) showed the highest resistivity of those materials measured which had been pre-exposed to mercury vapors at 538 C.

#### 3.2.1.5. Metal-to-Ceramic Seals

The electrical leakage resistance of an alumina (Lucalox) disk incorporated as part of a tantalum-alumina-molybdenum seal was determined in an atmosphere of tank argon at 850 C. This metal-to-ceramic seal employs a 95% zirconium-5% beryllium brazing composite. A 1/32" diameter molybdenum pin extended through and was sealed at the center of this disk. A 1/2" tantalum tube was sealed to the periphery of the 1/16" thick alumina disk. These metal-ceramic seals were found vacuum tight, prior to and after cycling to 850 C, with a helium-type leak detector. The measured electrical leakage resistance of this seal at 850 C in argon was  $9.7 \times 10^7$  ohms.

#### 3.2.1.6. Special Insulated Conductor

The electrical leakage resistance and volume resistivity of a commercially available insulation-conductor system from Advanced Technology Laboratory was determined at a temperature of 850 C in tank argon. The 1/8" diameter copper conductor of this system was electrically insulated by a 1/32" wall of

magnesia powder which was packed in a concentric 1/4" diameter sheath of 304 stainless steel. The measured leakage resistance and volume resistivity of the magnesia in this system are  $9.3 \times 10^6$  ohms per inch and  $1.4 \times 10^8$  ohm-inches, respectively. The electrical volume resistivity of magnesia as applied in packed powder form in this system is about 50 times higher than that previously reported for hot pressed magnesia at 850 C.

### 3.2.2. Testing in Mercury Vapor

#### 3.2.2.1. Test Capsule Construction

A number of Ceramaseal (Series 801 and 805) alumina to Kovar seals were successfully welded to a 1/2" diameter stainless steel (304) tube. These assemblies were charged with mercury, sealed, and submitted for electrical resistivity measurement over a 540 C temperature range. A second type of alumina to Kovar seal (Alite cable end seal) was sealed to a 1/2" diameter nickel tube. The capsule was then loaded with mercury and sealed. Although exposure to high temperature mercury vapor did not cause leakage at the ceramic to metal joint, the assembly ruptured at an apparently weak Kovar to Kovar joint inherent in the design of the seal. This joint is now being strengthened with a high melting brazing alloy (Incusil-Ag/Cu eutectic + 15% In) after which the test will be repeated.



#### 3.2.2.2. Electrical Leakage Resistance Measurements

Attempts were made to measure the electrical leakage resistance of five alumina-Kovar seals at a temperature of 538 C while one side of each seal was exposed to saturated mercury vapor. The alumina insulators were in the shape of a cylindrical tube about 1/2" long, 1/2" outside diameter, and 1/4" inside diameter. A Kovar flange cap with a threaded stud was silver-copper brazed to the top and a Kovar flanged cylinder to the bottom exterior portion of the alumina tube. The lower flange was heliarc welded or furnace brazed to 4-inch long stainless steel tubes which formed a reservoir for the liquid mercury. After charging the capsule with mercury, the steel tube was sealed with a steel "Swagelok" fitting. In every case mercury leaks developed, either in the Kovar flange or the alumina-Kovar seal, prior to attainment of the desired temperature conditions for electrical measurement. Reinforcement of the Kovar flange, shielding of the metal-ceramic brazing material, or use of higher corrosion resistant brazing material may provide a solution to the mercury leak problem.

Leakage occurred in a capsule using a Ceramaseal insulator at 300 C. The resistivity of the insulator at this temperature was  $5 \times 10^8$  ohm per square.

The last two capsules incorporating Alite C-500 cable end

seals started to leak about 400 C. Resistance values of  $4 \times 10^7$  ohms were measured prior to signs of leakage with an insulator temperature of 400 C and a mercury reservoir temperature of 380 C.

### 3.2.3. Testing in Potassium Vapor

#### 3.2.3.1. Ceramic to Metal Brazing Investigation

A review was made of the literature and several approaches were indicated:

- (1) Use active metals such as zirconium or titanium or their alloys to make a seal directly between the Lucalox and metals which have good resistance to potassium.
- (2) Coat ceramic with molybdenum; molybdenum-manganese, molybdenum-titania or other combinations which appear promising. Braze the metallized ceramic to the tube with the nickel base alloys which have good resistance to potassium.
- (3) Use a diffusion bonding technique in which the base metal of the tube is diffusion bonded directly to the alumina.

In general, the present work has consisted of making wetting tests with the various alloys on Lucalox. Tantalum was then brazed to the Lucalox with the most promising alloys and the specimen exposed to potassium vapors in stainless steel capsules at 850 C for 170 hours. Some preliminary tests were

made in attempt to coat Lucalox with Mo, Mo-Mn and MoTi. The procedure used was as follows. The wetting tests were made in vacuum at about  $10^{-5}$  torr. Small pieces of the brazing alloy (100 mg) were placed on small pieces of Lucalox tube. The alloy and Lucalox tube was then placed inside a ceramic tube which, in turn, was placed inside a molybdenum susceptor. The unit was pumped down to  $10^{-5}$  torr and the sample heated to about 1300 C and held for one minute. Micrographs of three of these specimens are shown in Figures 9, 10, and 11.

Another group of "T joints" was made between Lucalox and tantalum with the more promising alloys. These specimens were placed in stainless steel capsules with potassium and closed with a Swagelok. The units were then heated to 850 C for 170 hours.

A list of the brazing alloy compositions tested with results of the wetting tests and the potassium tests are given in Table 7.

The results of the tests indicate that most of the titanium-zirconium alloys wet pure alumina. Some, however, produce cracks in the Lucalox directly under the brazing alloy as shown in Figure 9.

All the brazing alloys exhibit rather poor resistance to potassium except as indicated below:

(1) 95% zirconia-5% beryllium. Four samples were tested.

Two separated in test and two remained intact but could be easily broken. Attack occurred at the interface on most specimens.

(2) 63% titanium-27% iron-10% vanadium - Test results doubtful - capsule leaked. Test will be repeated.

(3) 63% titanium, 27% iron, 10% Mo - Sample did not separate in test but could be separated with tweezers. Attack occurred at interface.

(4) 68% titanium -28% vanadium, 4% beryllium - Sample did not separate in potassium and could only be pried off the  $Al_2O_3$  with difficulty. A fine crack in the alumina caused fracture before potassium exposure.

The active metals are reported to wet alumina by a chemical reaction between the alumina and the active metal oxides. However, a typical layer of this reaction product cannot be definitely identified in the micrographs. Some alloys exhibit rather large columnar growth at the surface. Other alloys exhibit either a typical dendritic growth of metal at the interface or a bonded structure at the interface. All types appear to cause a general solution effect across the interface.

Although some cracking occurs in the Lucalox with several of the alloys, it is thought that a very thin layer of brazing alloy would produce smaller stresses in the Lucalox and minimize cracking. Such a configuration will be tested.

Lucalox to tantalum tube and sapphire to tantalum tube seals have been made during this period. The ceramic was 0.5 inch O.D. 1/16 and 1/8 inch thick, with a 0.030 inch hole in the center. These were brazed to the tantalum tubes with 95% Zr - 5% Be alloy. The tubes were 0.5 inch O.D., so that they had to be flared out to obtain a satisfactory slip-fit of the ceramic disk. The wall thickness of the tantalum was 0.020 inch. A 0.020 inch Ta wire was brazed into the center hole for lead-in.

Out of seven attempts, two brazed slots were satisfactory; i.e., they were vacuum tight.

#### 3.2.3.2. Electric Leakage Resistance Measurements

No electric leakage resistance measurements were made in a potassium vapor environment during this quarter due to unavailability of a suitable test capsule.

### 3.3. Wire Coating Methods

#### 3.3.1. Plasma Sprayed Coatings

Various oxides were plasma sprayed onto several substrates to optimize processing parameters and obtain samples for adherence tests and metal vapor exposure tests. A list of the materials plasma sprayed and substrates on which they were sprayed are given in Table 8.

Satisfactory coatings were obtained with all materials used. The substrate when properly prepared by solvent cleaning and light sandblasting was easily coated in all cases.

Samples of each oxide on an Inconel substrate were subjected to thermal cycling to 850 C. The samples were heated from room temperature to 850 C over a period of 1-1/2 hours and allowed to cool in still air to room temperature. No evidence of separation of the coating from the substrate was found on any of the samples.

The plasma coated oxides on Inconel substrates were subjected to potassium environment tests. The results of these tests are given in Table 1. The alumina (Metco 101) was attacked, probably through the titania additive in the material. The purer alumina (Metco 105) seemed to be intact and unaffected

but separated from the Inconel substrate. The hafnium-free zirconia, Metco XP-115, was virtually unaffected and no change in appearance occurred. The calcia stabilized zirconia, Metco 201, remained as an insulating film after exposure but appeared from weight loss and color change to have lost the calcia.

### 3.3.2. Coating Preformed Coils

A system was devised and built for holding preformed coils of wire in a fixture which could be continuously rotated during the plasma spraying operation. Using this equipment, a preformed coil of nickel clad copper was plasma coated with alumina (Metco 105). The above coil was used for initial coating tests because it was available from another project and was very similar to the statorette coils. A photograph of the coated coil is shown in Figure 10. The coating generally was very uniform and adherent. One small area which had excessive build up of the oxide cracked when the coil was compressed. This crack appeared at the apex of the acute angle on the end of the hexagonal coil. Additional coils preformed to the exact size to be used on the statorettes were constructed using AWG No. 8 nickel clad silver wire. These coils were coated with alumina (Metco 105) and again had a very uniform, thin coating. When these coils were compressed, no visible cracking or loss of coating occurred. Better adherence is expected on

these latter coils since the ends of the coil have a uniform radius and therefore do not concentrate the bending stresses at the ends as do the initial evaluated hexagonally shaped coils.

A silica free fusion coating has not been obtained even at temperatures in excess of the maximum temperature allowable for the conductor material. Very brief exposures to 1400 C was attempted to obtain fusion but without success. Even though this temperature was 450 C above the allowable conductor temperature, the short duration of the exposure allowed the coating to reach a high temperature without the conductor exceeding its maximum temperature.

The fusion coating obtained and previously reported was found to contain a high percentage of titania. Although this coating is an insulator, its properties at 850 C would not be as satisfactory as coatings without titania.

Very good coatings have been obtained using fine alumina in a colloidal zirconia solution. The coating, when dried and fired, is adherent, uniform and will not rub off. This coating has been successfully applied to stainless steel, Inconel and nickel substrates. Application to stainless steel has resulted in the



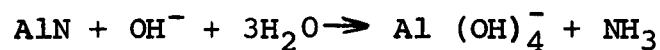
coating popping off or having blisters in certain areas after firing at 1000 C. Microscopic examination of the fired coating in the blistered areas has shown the formation of an oxide coating on the stainless steel causing this blister effect. Firing in an inert atmosphere would eliminate the problem.

Subsequent overcoating of the alumina-zirconia coating with ammonium zirconyl carbonate has produced a very smooth, abrasion resistant surface. Unfortunately, this overcoating on refiring produces enough shrinkage to form cracks in the coating.

### 3.3.3. Formation of Aluminum Nitride

#### 3.3.3.1. General

It has been reported by Th. Renner<sup>1</sup> that films of aluminum nitride can be deposited on a graphite disc by passing vapors of the  $\text{AlCl}_3 \cdot \text{NH}_3$  addition compound over the disc heated to 1000-1700 C. Films deposited at the higher temperatures have better formed crystal structures, are whiter, and are more chemically stable than those formed at lower temperatures. Renner states that white AlN is hardly attacked by hot water, dilute or concentrated HCl,  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$  or aqua regia. It is decomposed by hot concentrated NaOH as represented by the equation:



#### 3.3.3.2. Preparation of Source Material

This addition compound was prepared by passing anhydrous ammonia over  $\text{AlCl}_3$  at about 150 C until the gross composition of the reaction mixture was  $\text{AlCl}_3 \cdot 3\text{NH}_3$ . This is a mixture of compounds containing various ratios of aluminum chloride to ammonia. It was then heated to 350 C for several hours. In this treatment, excess ammonia is driven off giving a composition corresponding to  $\text{AlCl}_3 \cdot \text{NH}_3$ . This was purified by distillation at 170 C/0.02 torr. The product is a snow white solid with a melting point of about 125 C. It is sensitive to moisture but is quite thermally stable up to its normal boiling point of 420 C.

#### 3.3.3.3. General Experimental Procedure

The apparatus consisted of a straight 30mm diameter quartz tube in which was placed one porcelain boat containing  $\text{AlCl}_3 \cdot \text{NH}_3$  and another, about 6 inches farther downstream, containing the substrate. A resistance heater was used to heat the portion of the tube containing the source material while the substrate was heated with a radio frequency coil which surrounded the tube at the location of the substrate. All depositions were carried out in a continuously pumped system at a pressure of about  $10^{-2}$  torr. Time of a typical run was about 4 hours.

#### 3.3.3.4. Graphite Substrate

Vapors of the source material were passed over graphite rods heated at 1000 to 1550 C. The temperature of the source material was varied from 125 to 200 C corresponding to vapor pressure of about  $10^{-2}$  to  $10^{-1}$  torr. At this higher temperature, the material showed little tendency to deposit on the graphite, but at 1000 C, well bonded continuous insulating films were obtained. Unsupported films of the coating were obtained by heating a coated graphite rod which had been cut in half for several hours at 700 C. The rod was partially burned away so that it was easily separated from the film. Portions of this film were soaked in water and dilute HCl for several weeks with no apparent deterioration. A very small amount of gelatinous precipitate formed in a sodium hydroxide solution in which a piece of the film was soaked for 3 weeks. Elemental analyses are being determined on the film.

An x-ray diffraction pattern was obtained on a portion of the coating which chipped from an unusually heavily coated end of a graphite rod. The rod and separated disc are shown in Figure 13. The diffraction pattern corresponds to a hexagonal wurtzite crystal structure which aluminum nitride is known to have. The pattern is shown in Figure 14.

An aluminum nitride coated graphite rod and a piece of unsupported AlN film have been placed in test capsules for potassium

vapor exposure tests.

#### 3.3.3.5. Molybdenum and Tungsten Wire Substrates

A 20 mil molybdenum wire coil was heated to 935 C in the presence of  $\text{AlCl}_3 \cdot \text{NH}_3$  vapor. A thin adherent insulating film was formed on it. A 20 mil tungsten wire coil was heated to 970 C in the presence of  $\text{AlCl}_3 \cdot \text{NH}_3$  vapor. The coating was about 1 mil thick. Numerous cracks were visible in it.

#### 3.3.3.6. Oxalloy 28 Wire Substrate

Oxalloy 28 is a stainless steel clad copper wire of about 50 mil diameter. Several attempts were made to apply aluminum nitride to coils of this wire but all were unsuccessful. Any coating which was formed chipped off as the wire cooled. A photograph of one such coil is shown in Figure 15. During runs with Oxalloy as the substrate, a cloudy yellow film formed on the quartz tube. This might indicate that the source material is reacting with the wire giving a surface to which the AlN will not adhere. To determine whether a metallic chloride was being formed on the wire surface as a result of reaction with  $\text{AlCl}_3 \cdot \text{NH}_3$ , an Oxalloy coil, which had been exposed to the addition compound at 900 C, was put into water. However, a test for chloride ion in the water was negative. While the coil was in the water, a loose rusty sediment formed on it. This did not occur in a similar coil which had not been exposed

to  $\text{AlCl}_3 \cdot \text{NH}_3$  at 900 C.

#### 3.3.3.7. Plasma Sprayed Alumina Substrate

An Oxalloy 28 wire coil which had been plasma sprayed with about 25 mils of alumina was exposed to  $\text{AlCl}_3 \cdot \text{NH}_3$  vapors at 875 C. Two anticipated troubles with this experiment did not materialize. It was feared that the difference in the thermal expansion of the wire and alumina would cause spalling of alumina at 875 C. This did not occur. Another anticipated problem was that the interface between the alumina coating and the wire would make it difficult to bring the alumina up to the temperature. However, no difficulty was encountered in heating the alumina surface to 875 C. Apparently it is well bonded to the wire.

As the vapors of  $\text{AlCl}_3 \cdot \text{NH}_3$  were brought into contact with this alumina coating, areas of a green film formed on the alumina. These were somewhat conductive. They seemed to originate at cracks in the alumina coating. Figure 16 is a photograph of this coil. Another coil with a thinner alumina coating, without apparent cracks, was also exposed to  $\text{AlCl}_3 \cdot \text{NH}_3$  vapor at 875 C. A fairly uniform dark grey coating covered the alumina. It was not conductive. Figure 17 shows this coil alone with another alumina coated coil which has not been exposed to

$\text{AlCl}_3 \cdot \text{NH}_3$  vapor. It was submitted for exposure to potassium vapor.

#### 3.3.4. Formation of Boron Nitride

Some effort was devoted during this quarter to preparing coated substrate for evaluation in potassium vapor tests. The vapor phase pyrolysis of B-trichloroborazole was carried out in the vacuum set up described in the second quarterly progress report. Work was directed at getting continuous and hole-free boron nitride coatings on the substrates, stainless steel sheathed copper and graphite rod. Only the rod and wire samples which showed the most continuous coatings were used in the potassium vapor tests.

The reason for the selection of graphite as a substrate was that it is fairly compatible with the BN film and that it is highly susceptible to attack by potassium vapor at 850 C. Previous tests showed that graphite rod exposed to the metal vapor was completely reduced to a powder. The condition of a filmed graphite rod at the completion of the potassium vapor exposure tests would give some insight as to the ability of the film to provide to provide protection against potassium attack.

Results of the exposure of the filmed substrates to potassium

vapor at 850 C for 172 hours . . . indicate some degree of resistance to the alkali metal attack.

Figure 18 shows the appearance of several graphite rods. The top rod is untreated, the center rod is filmed with BN but has no other treatment, while the bottom rod (sample RB24-1) had been exposed to potassium vapor at 850 C for 172 hours. The rod still retains a good portion of its mechanical strength and manifests alkali metal attack predominately at the surface, which has a flaked appearance. After 6 weeks exposure to air, this flaked surface acquired a white grey cast. The rest of the rod remained black in color. This color change might be due to hydrolysis of the BN film remaining on the rod. There was only a slight tendency of the rod to powder. Taking into consideration the fact that the BN coating on the rod was not perfectly continuous and hole-free to start with, there is some indication that the film afforded the graphite a measure of protection.

Figure 19 is a micrograph of a BN film deposited on a graphite rod. This compares well to a photomicrograph of a BN film obtained by Patterson et al<sup>(2)</sup> except that in this case the grains are somewhat larger than that obtained by Patterson.

Figure 20 is a micrograph (200X) of RB24-1, the boron nitride

film on a graphite rod after a 172 hour exposure to potassium vapors at 850 C.

Few deductions can be drawn from the run on stainless steel clad copper wire coil (sample RB371). The stainless steel sheathing appeared intact and had no obvious chemical attack. The film was opaque white to grey in color and is very easy to wipe off the conductor. The film remaining on the coil is nonconducting. Figure 21 shows the coil after exposure to potassium vapor at 850 C for 172 hours. Figure 22 shows a boron nitride coated stainless steel clad copper wire. The film, which ranges from transparent to opaque white in samples, is transparent in this case.

X-ray results on the films of some previously run samples have been received. Table 9 lists the samples, substrates and substrate temperatures during the pyrolysis of the trichloroborazole. The d values for these samples are listed in Table 10 along with the d values of BN and graphite from the ASTM index. All patterns were taken (with the exception of S13M) in 114.6 mm Debye Scherrer powder cameras using nickel filtered copper radiation. Sample RB241-B was a piece of the film from RB24-1-A which was given an additional 3 hours heating in air at 980 C. Sample R53R-2 was a fluffy white deposit on the boat holding the tungsten wire coil (sample R53R-1). The boat was relatively



cool compared to the wire coil.

The x-ray diffraction patterns for the samples are shown in Figures 23, 24, 25, 26, and 27.

The samples are of a low order of crystallinity since bands were formed instead of sharp lines. The only exception is sample R53R-2 which is amorphous. The absence of impurity lines does not exclude the presence of impurities but merely indicates they are not present in a measurable degree or that they are masked by the broad sample lines. Except for line width and sharpness, all patterns are identical except R53R2 which is amorphous. Because of the similarity in structures of BN and graphite, all samples with graphite substrate should be interpreted with caution. Sample S13M was identified as hexagonal boron nitride. These x-ray results agree with those described by Patterson.<sup>(2)</sup> The iron oxides appearing in sample RB11X1 are from the momentary failure of the vacuum system.

Infrared spectra of several samples from different substrates were secured. Hexagonal boron nitride has absorption peaks at 7.2 and 12.2 microns. The spectra from the samples can be divided into 3 groups.

A) Samples R29I (platinum substrate at 1300 C), R4401-1

(Inconel substrate at 1155 C), RB271D, and RB271C gave a very broad maximum in the 8 and 9 micron region and a broad absorption peak at 12.5 microns. Samples RB27-1-D and RB27-1-C were fragments of a spalled film obtained by heating a graphite rod substrate at 1375 C in trichloroborazole vapors, cooling, removing the rod, rotating and revolving the rod 180° and reheating in  $\text{Cl}_3\text{B}_3\text{N}_3\text{H}_3$  vapors. The second coating flaked off the rod and was used for samples. It should be noted that this coating was formed at a lower temperature than indicated at the surface of the rod. RB27-1C was submitted with no further treatment. RB27-1D was heated in air for 3 hours at 980 C. The infrared spectra data on the above samples are shown on Figures 28 and 29.

B) Samples R50Q-1 (film from tungsten was substrate at 1450 C), S9F (tungsten rod substrate at 1050 C and S53R-2) a powdery white deposit in front of a boat containing a tungsten substrate at 1950 C gave an absorption peak at 7.2 microns and a broad absorption peak at 12.5 microns. Infrared spectra data on these samples are shown in Figures 29 and 30.

C) Sample R53R-1 (deposit on a tungsten wire coil substrate at 1950 C) has absorption peaks at 7.2 and 12.2 microns and a broad absorption peak at 12.5 microns. Infrared spectra data are shown in Figure 30.

The infrared spectra indicate the samples to be boron nitride but of varying degrees of crystallinity. Sample R53R-1 is the only one having hexagonal boron nitride to an appreciable degree according to the infrared results. The higher temperatures of the substrate tend to give boron nitride of a more hexagonal crystalline nature. This is borne out by the x-ray data which shows a slight sharpening of the lines with increasing substrate temperature.

The majority of the BN filmed substrates gave off an odor of ammonia after being exposed to air for some time. There is a basic reaction with moist litmus paper held over the mouth of the vials. Poaszus<sup>(3)</sup> reported that on extensive boiling with water or acid solutions, boron nitride hydrolyzes to  $\text{NH}_3$  and boric acid. Furthermore, it has been reported in the literature that the chemical stability of BN is dependent on the temperature at which it is produced. Boron nitride produced at 130 C is easily decomposed by moisture<sup>(4)</sup>, while BN made at 800 C will begin to decompose after being exposed to air a short time, and that heated to about 1000 C showed only slight signs of decomposition after air exposure<sup>(5)</sup>. Boron nitride heated at 1250 C is not attacked by boiling water<sup>(6)</sup>. Since our films are formed by induction heating of the substrate, the films are formed at a lower temperature than that indicated at the surface of the substrate.

Chemical analysis has not been used because of the small amounts of the samples involved, the sample instability and analytical difficulties.

### 3.3.5. Mass Transport of Alumina

The principles of densification of porous coatings by mass transport of material from the outer layers of an insulating coating to the layers immediately adjacent to the electrical conductor were discussed in the second quarterly report (Sec. 4.3.7). A preliminary experiment was described in which material, allegedly high purity alumina, was transported a distance of 15 cm in a quartz tube.

Analysis of the material transported toward the cooler side in the thermal gradient system involving the equilibrium



showed it to be  $\text{Al}_2\text{O}_3$  in form of rather large crystallites. The crystalline portion comprised over 90% of the sample. The transported material contains 0.20% Fe whereas the source material only contains 0.03% Fe. The transported material also contains a residual 1.2% of Cl.

The  $\text{Al}_2\text{O}_3$  used as source material had changed color to dark gray after the transport experiment. This color was traced to contamination with carbon of unknown source. Our conclusions are:

(1) transport of  $\text{Al}_2\text{O}_3$  to a desired location does take place at a useful rate, (2) the transported  $\text{Al}_2\text{O}_3$  is deposited largely in the crystalline form (which is probably necessary, for resistance to potassium vapor), (3) Iron present as an undesirable impurity is transported even more rapidly than  $\text{Al}_2\text{O}_3$  and probably by a similar mechanism, (4) The Cl found can be removed by after bake of the sample, (5) An unknown, probably organic, contaminant is present in the system and acts as a source of carbon.

Apparatus was set up to try transport of  $\text{Al}_2\text{O}_3$  on a wire mock up. The simulated wire was tantalum to prevent any attack by the HCl environment. A porous  $\text{Al}_2\text{O}_3$  coating was applied to the Ta rod from a slurry. The baked out coating on simulated Ta wire popped off on cooling due to oxidation of the Ta in the furnace. The  $\text{N}_2$  and  $\text{A}_2$  cover gases were apparently contaminated. Re-runs produced adherent, porous  $\text{Al}_2\text{O}_3$  coatings.

Investigation of this technique was terminated in late September to concentrate effort on more promising solutions and more pressing problems such as the metal-ceramic seal for potassium vapor applications.

### 3.3.6. Formation of Alumina by Thermal Decomposition

With the intention of eliminating the carbon formation problem

experienced with aluminum isopropoxide (see Sec. 4.3.5. Second Quarterly Report), a quantity of aluminum methoxide was prepared. This compound has a much lower carbon content and does not contain the relatively easily cracked isopropyl group. A disadvantage found with the methoxide is its very low volatility. To pass aluminum methoxide vapors over a hot substrate, it will be necessary to incorporate into the deposition apparatus, a vacuum source capable of  $10^{-5}$  torr.

Investigation of this technique was terminated to concentrate effort on the AlN formation investigation.

### 3.3.7. Discussion of Wire Coating Methods

In consideration of the technical problems in coating conductors and the results of the potassium environment tests the choice of usable conductor insulations is very limited. The plasma sprayed zirconia appears to be the best coating as far as resistance to potassium vapor but does not have as high an insulation resistance at high temperatures as the other insulating materials. Plasma sprayed alumina is another candidate and may prove successful on round conductors. The alumina would be a better electrical insulator but adherence to the metal substrate during exposure to potassium vapor has been a problem. The fusion coating of alumina and zirconia has failed to pass the potassium vapor test even though the basic components

are resistant to potassium vapor.

Plasma coating of preformed coils seems to be very feasible for making coils for the statorette environment tests. The present information suggests that the plasma sprayed zirconia coating is the best.

A convenient method of applying alumina to wire coils is by plasma spraying. However, these coatings are usually quite porous. It was felt that a film of vapor deposited AlN might fill the pores of a plasma sprayed coating. Or, looking at it from another angle, a thin plasma sprayed alumina coating on Oxalloy wire might provide a suitable surface onto which AlN could be applied. Success in this area would help in our inability, to date, apply AlN to Oxalloy wire.

If the particular form of AlN which is being formed under our experimental conditions proves to be unstable in the presence of alkali metal vapor, it might be exposed to air or oxygen at high temperature to convert the surface to  $\text{Al}_2\text{O}_3$ . The alumina surface, which is known to begin to form at 700 C, protects the bulk of the crystal from further oxidation, much as in the case of the metal itself.

Experimental conditions are still being varied in an attempt

to apply AlN directly to Oxalloy 28 wire. Optimum conditions for vapor depositing AlN in the pores and on the surface of plasma sprayed alumina are also being investigated.

The advantages of formation of BN coatings by vapor phase pyrolysis on heated substrates are that denser, more hole-free films can be formed. No binder is necessary. When induction heating is used to heat the samples, the deposition occurs directly on the substrate. Heating the sample by means of a resistance furnace causes depositing in the whole heated zone. One disadvantage in induction heating is that the film is formed at a lower temperature as it progresses out from the surface of the substrate. This would require the sample to be annealed later in a resistance furnace to increase the chemical stability of the coating.

The elevated temperatures required to achieve a chemically stable coating of BN are too high for the comparatively low melting points of the conductors, therefore, the vapor phase formation of BN on the wire conductors now under consideration is not practicable.

#### 3.4 Potting Compounds

Work on potting compounds during this quarter was limited to preparation of test samples and evaluation of the resistance



to attack by potassium vapor at 850 C.

The samples of potting compound exposed to the potassium vapor are listed in Table 1 as PK-1 through PK-5. The sample PK-1 was zirconia bonded magnesia. PK-2 and PK-4 were Sauereisen cements No. 30 and 9 respectively. The PK-3 and PK-5 potting compounds were Westinghouse compositions using phosphate bonds. The last four materials had been tested prior to this program and were, in our opinion, the best available for evaluation in potassium. However, it was believed that most bonding materials for potting compounds would be attacked to some degree. The compound PK-1 was developed and used because it contained only known potassium resistant materials. (At least in their dense fused state). All samples failed by disintegration, probably from attack of potassium on the bonding material.

### 3.5. Conductors

During the quarter orders were placed with Sylvania for lengths of AWG No. 8 nickel clad and Inconel clad silver wire. A three hundred foot length of nickel clad (20%) silver wire was received early in the quarter and has been used in the plasma spray and AlN formation work. A six hundred foot length of Inconel clad silver wire was scheduled for shipment from Sylvania by mid-October; however, the shipment date has now been moved to late December.

#### IV. PROGRAM FOR NEXT QUARTER

##### 4.1 Corrosion Studies

Additional exposures will be run during next quarter on coated samples, brazing alloys and metal-ceramic seal assemblies. One insulation material that will be exposed to potassium vapor is the 99% alumina, 1% magnesia, trace silica insulation used by the Alite Division in one of their lead-in assemblies. The material is identified as A-610 by Alite.

##### 4.2 Electrical Tests in Argon

Tests in argon will be made to obtain pre-metal vapor exposure leakage resistance values on lead-in seal combinations and insulated wire specimens.

##### 4.3 Electrical Tests in Metal Vapor

When suitable ceramic seals are found, the electrical resistivities of selected materials will be measured while suspended within the sealed capsules containing mercury vapor at 538 C or 850 C.

Our first efforts will be to establish the electrical leakage resistance of a tantalum-Lucalox-tantalum capsule and a tantalum-sapphire-tantalum capsule each sealed with the 95% zirconium - 5% beryllium brazing combination.

Trials will be made to wet alumina directly with molybdenum or molybdenum-titanium so that final joints could be made with the conventional nickel base brazing alloys or perhaps the pure metals, nickel or iron which have good resistance to potassium at 850 C.

Tests will also be made with Lucalox disks coated with molybdenum-manganese or molybdenum by an outside supplier, without degradation of the alumina.

Other trials will be made to obtain diffusion bonded seals between Lucalox and tantalum. Other tests are to be made to coat the active metal joints and interfaces with potassium resistant materials.

Several alumina tantalum tube seals have been made with the zirconium 5% beryllia alloy and the titanium vanadium beryllium alloy. Tubes made with ~~the~~ zirconium beryllium alloy have been leak tight and are to be tested. One tube has been electroplated with nickel on the inside to minimize attack on the brazing metal and the interface.

#### 4.4 Coating Studies

##### 4.4.1. Plasma Sprayed Coatings

Preformed coils of nickel clad silver wire will be plasma sprayed

with zirconia. Samples of magnesia and strontium zirconate on Inconel substrate will be prepared and evaluated in the potassium environment. If these latter coatings are sufficiently resistant to potassium vapor, preformed coils will also be coated.

#### 4.4.2. Fusion

The use of a fusion coating on the present wire will not, in light of the data from the potassium exposure tests, be practical. No further work will be done on the present coating.

#### 4.4.3. Aluminum Nitride

Since the aluminum nitride was applied successfully to some substrates further efforts will be directed towards application to Inconel and nickel clad silver wires. The possibility of heat treating the aluminum nitride coating in air to convert it to the oxide will be investigated.

The coating that appears to be most resistant to the potassium vapor and best electrical properties will be applied to preformed coils of nickel clad silver for evaluation in statorettes.

## V. REFERENCE LIST

1. Th. Renner, Zeit fur Anorg and Allgemeine Chem. 298, 23, 1959.
2. R. J. Patterson, R. D. Humphries, and R. R. Haberecht, "Thin Films of BN", presented at the National ECS Meeting, Pittsburgh, April 18, 1963.
3. E. Poaszus, "Process of Making Solid Bodies from Nitrides", United States Patent 1,337,264, April 20, 1920.
4. A. Stock and M. Blex, Ber. deut. chem. Gessell., Vol. 34, 1901, p. 3039.
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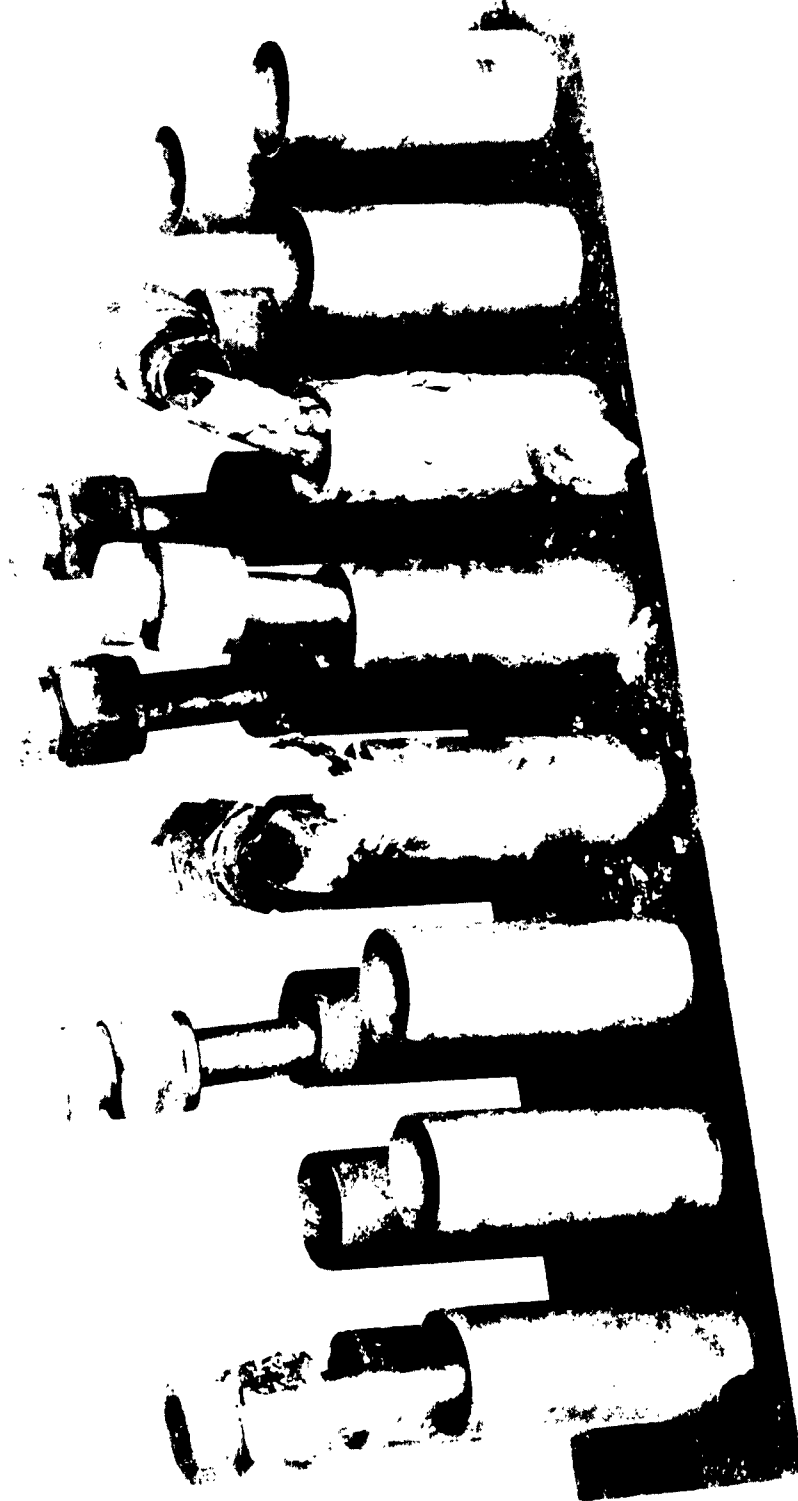


Figure 1 - Potassium exposure capsule holder  
immediately on removal from aging  
oven.

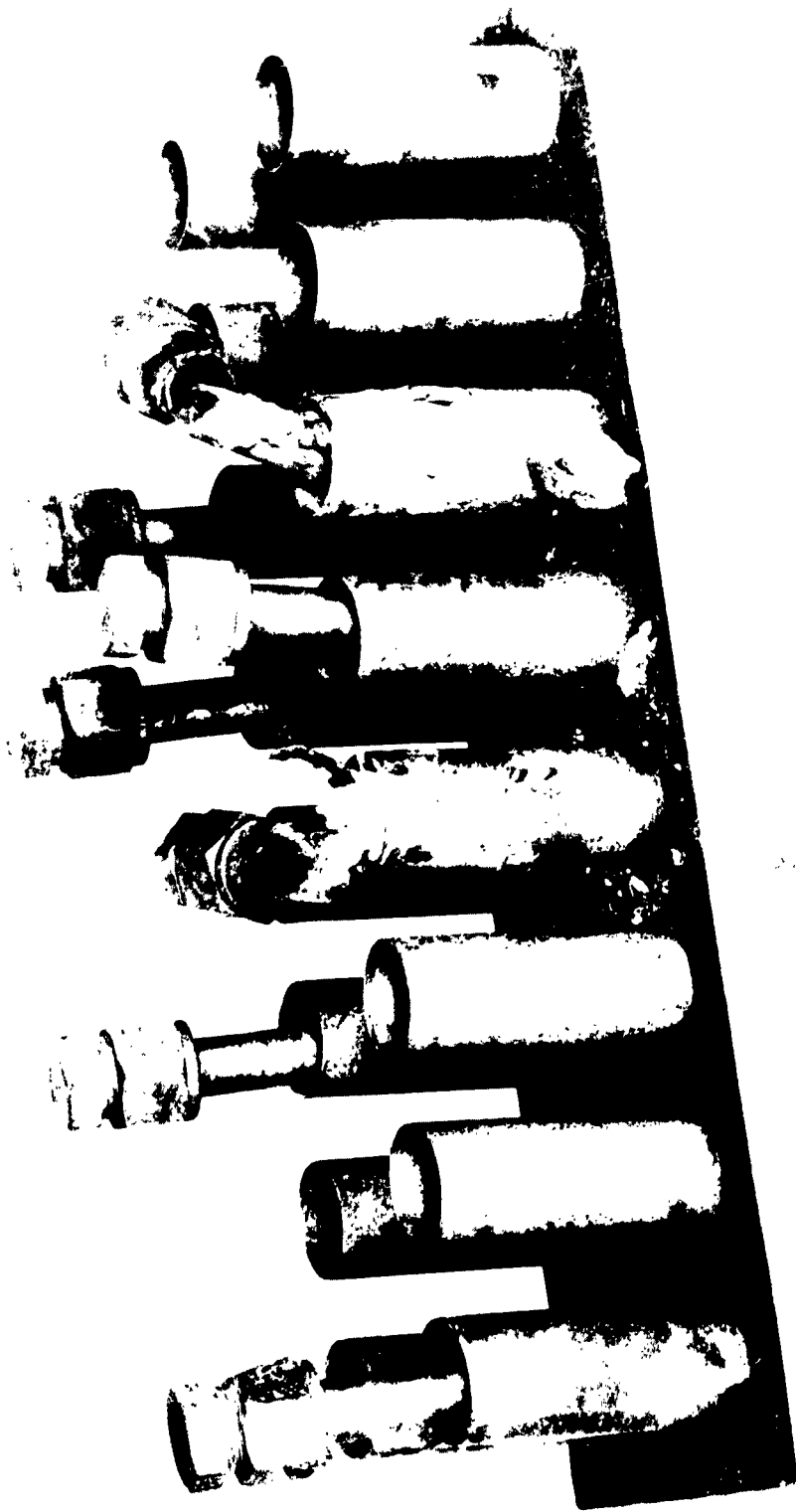


Figure 1 - Potassium exposure capsule holder  
immediately on removal from aging  
oven.

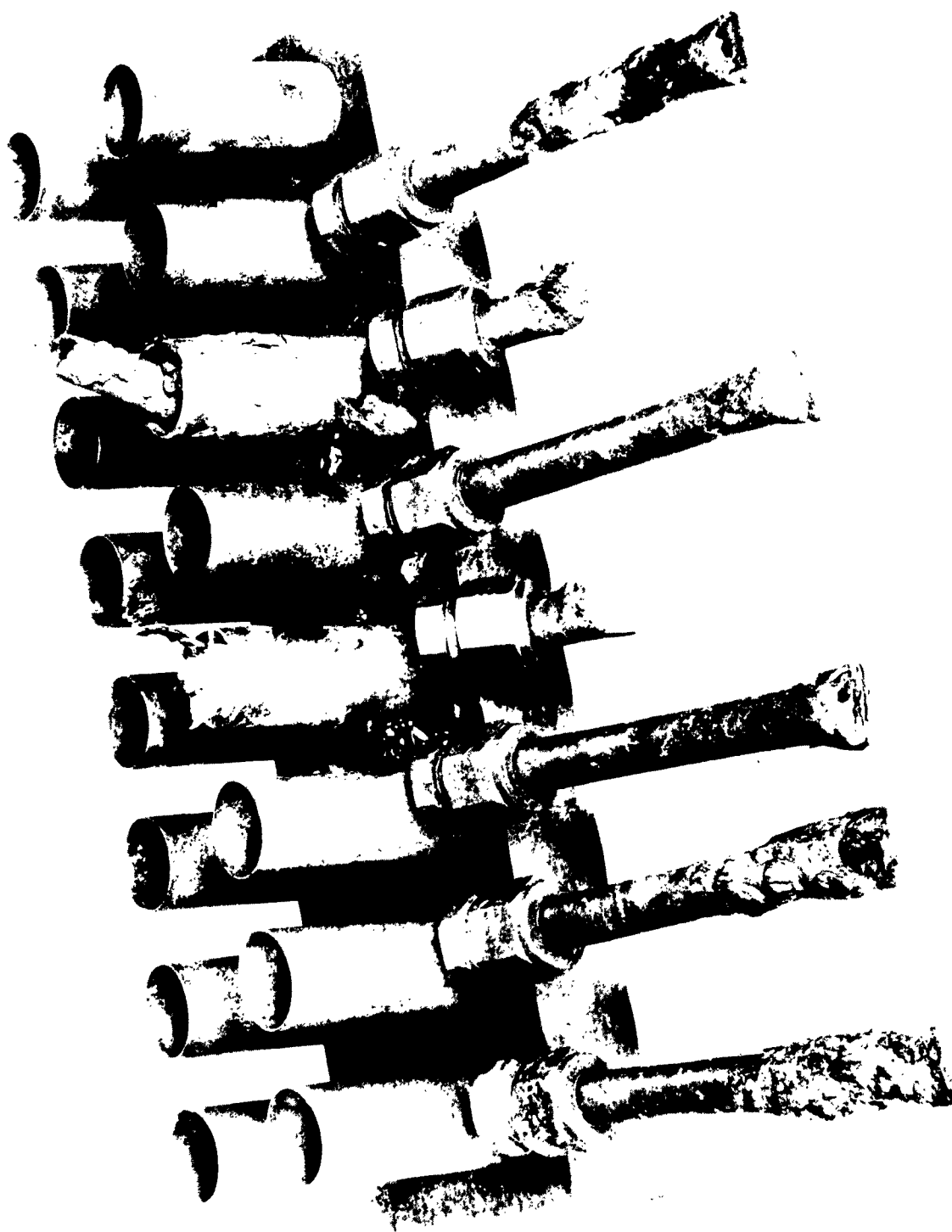


Figure 2 - Potassium exposure capsules  
removed from holder.



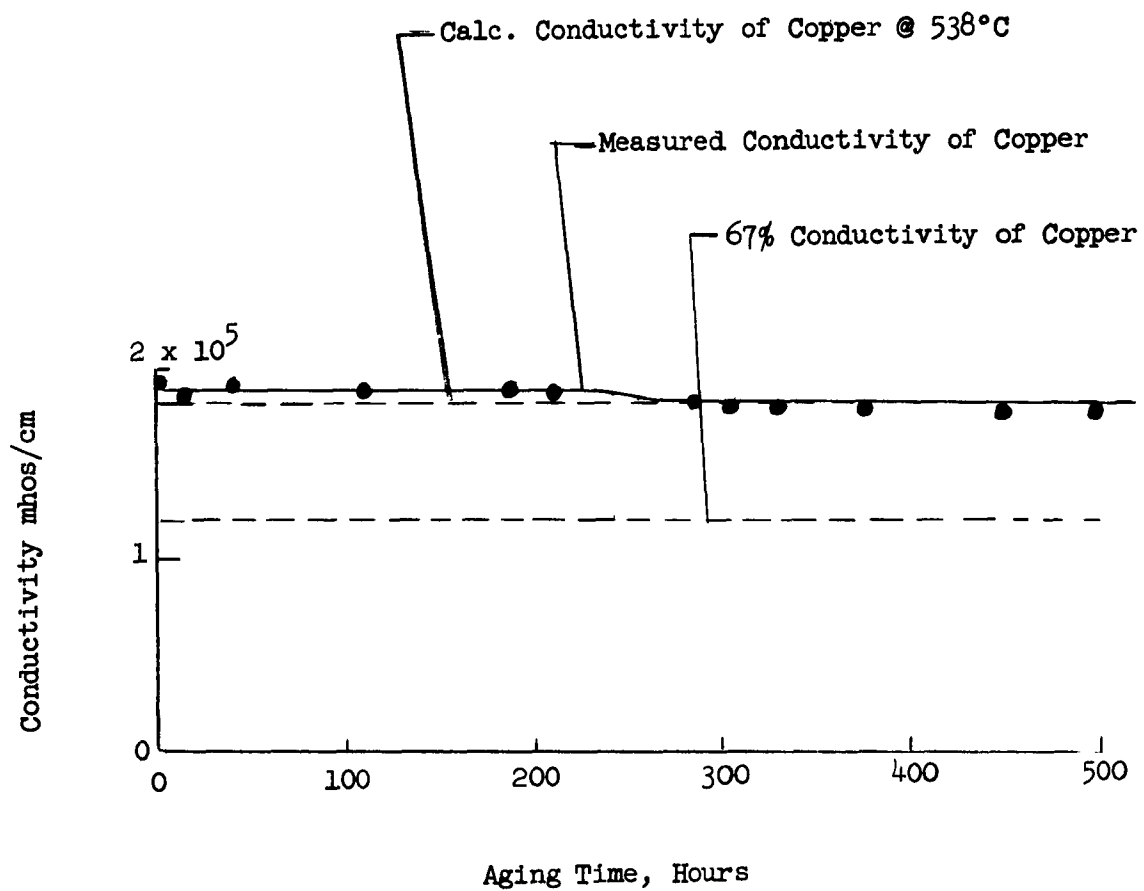


Figure 3 - Temperature Aging-Conductivity Characteristics of Copper Wire in Tank Argon at 540°C.

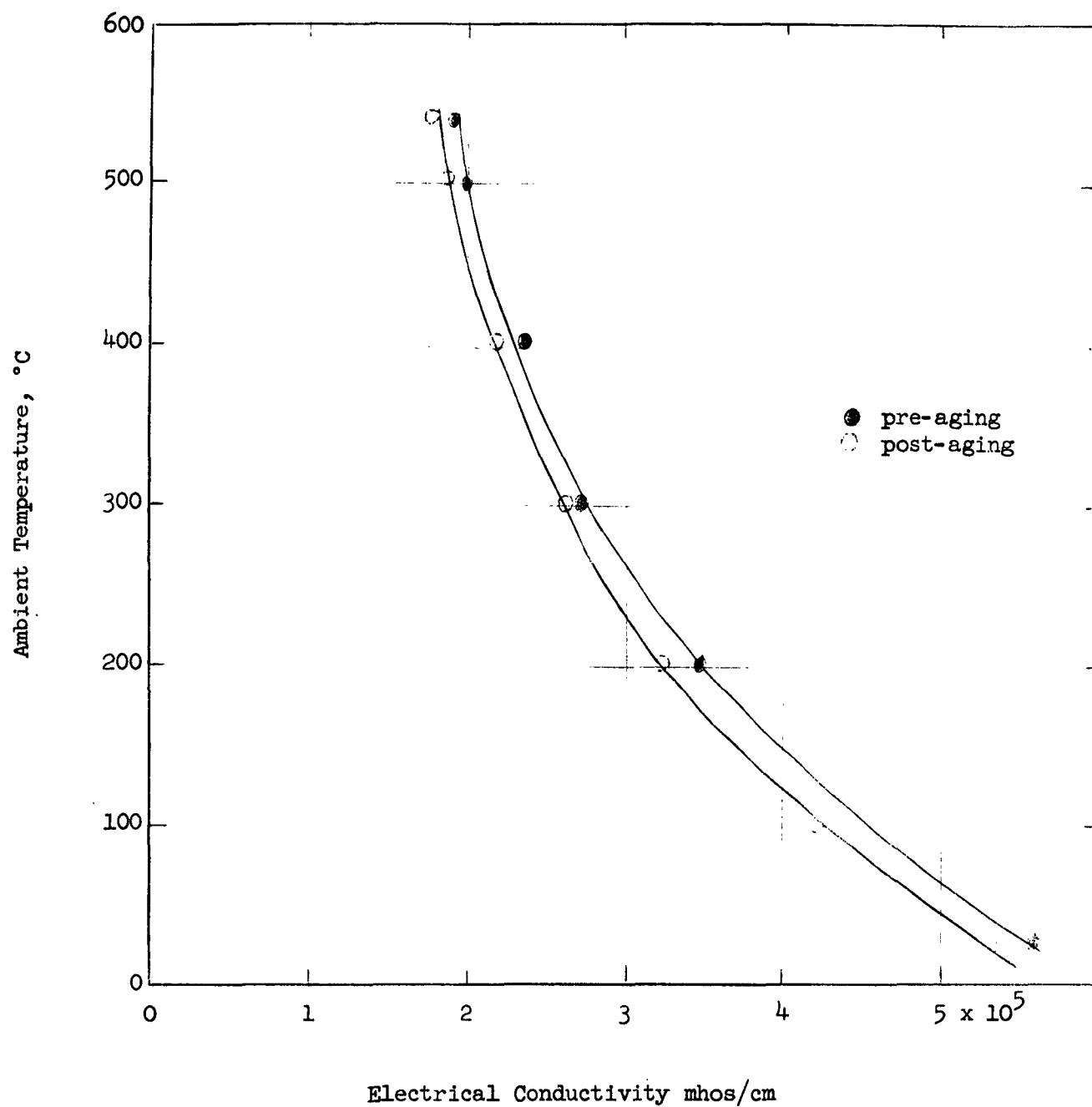


Figure 4 - Pre- and Post-Aging Characteristics of Bare Copper Wire at Temperatures to 540°C In Tank Argon.

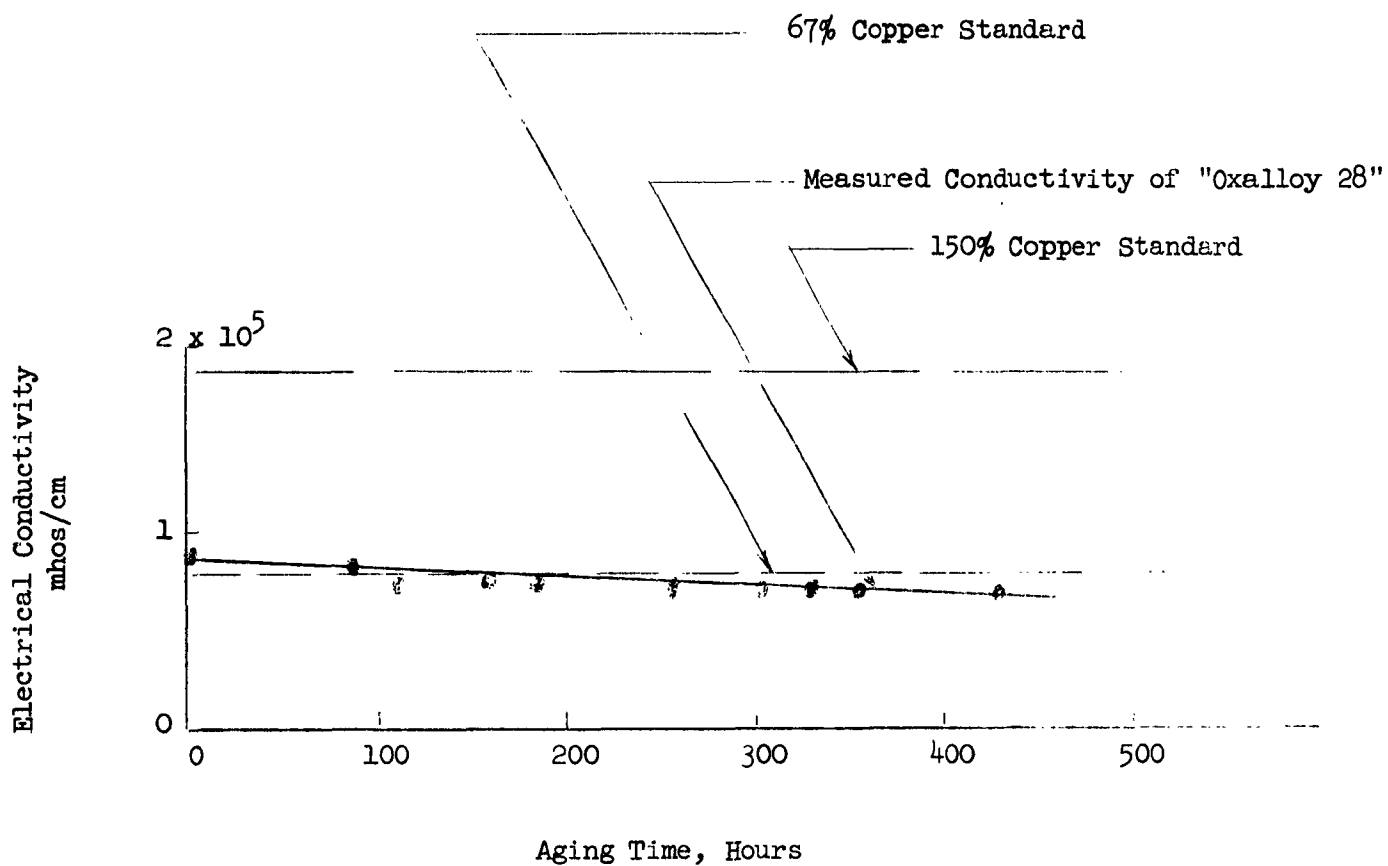


Figure 5 - Temperature Aging-Conductivity Characteristics of "Oxalloy 28" in Purified Argon at 850°C.

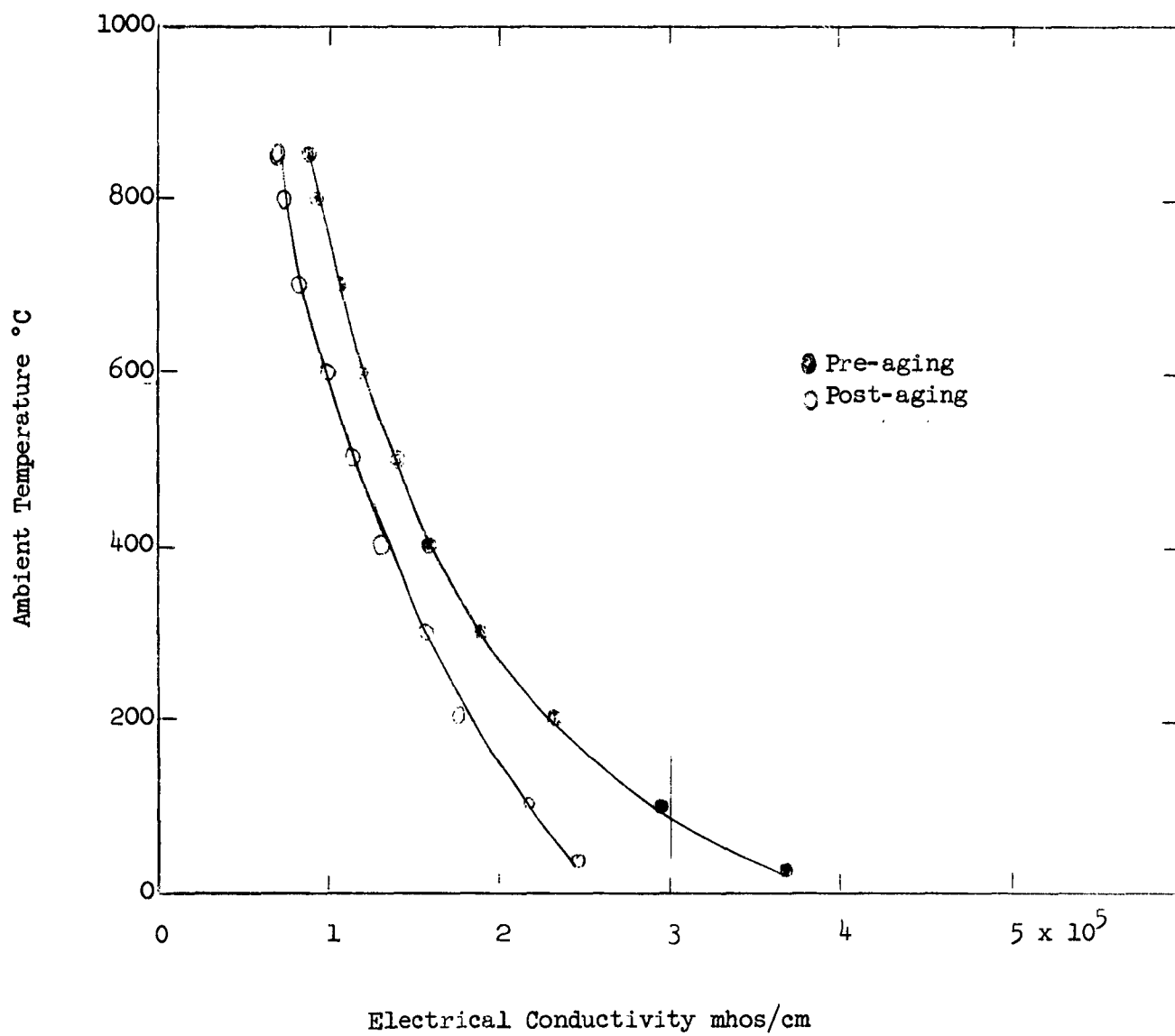


Figure 6 - Pre-and Post-Aging Temperature-Conductivity Characteristics of "Oxalloy 28" at Temperatures to 850°C in Purified Argon.

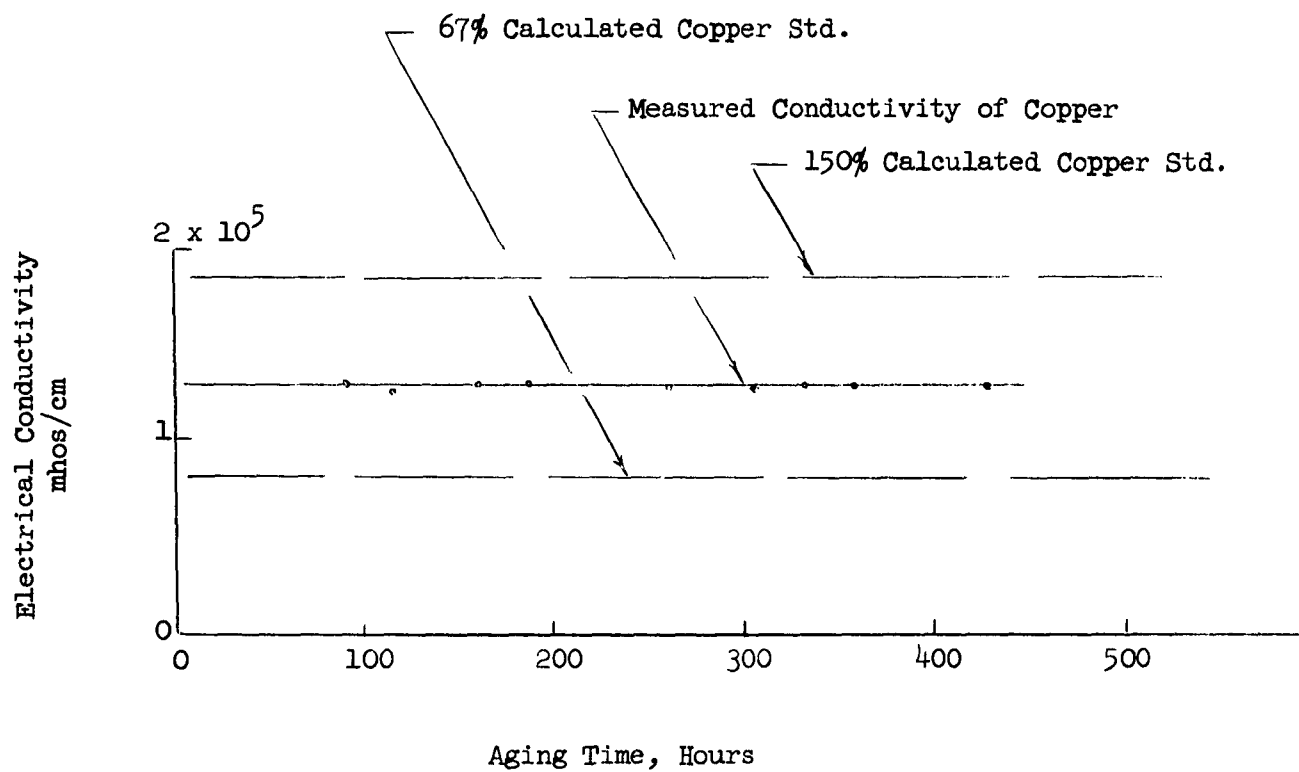


Figure 7 - Temperature Aging-Conductivity Characteristics of Copper Wire in Purified Argon at 850°C.

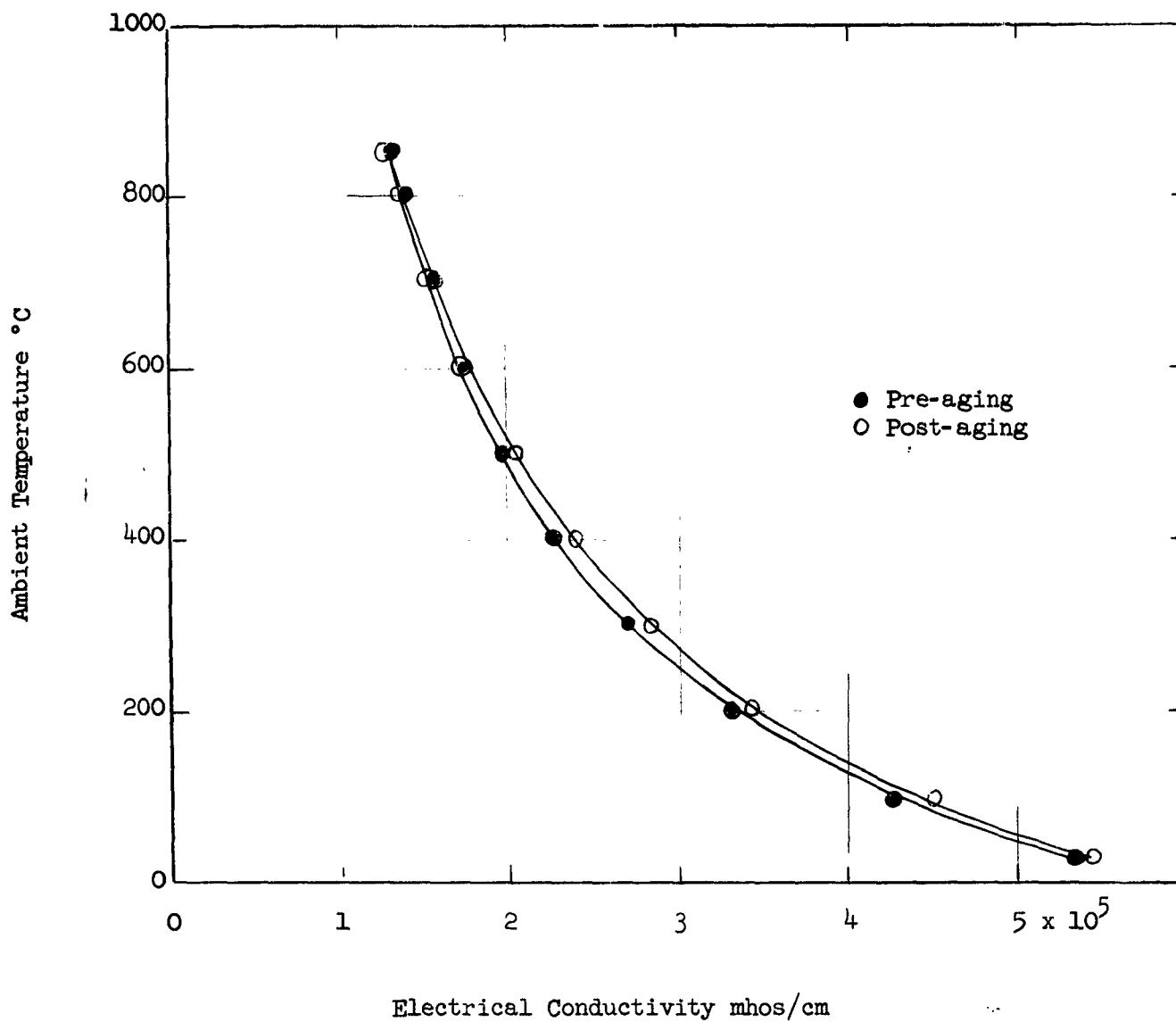
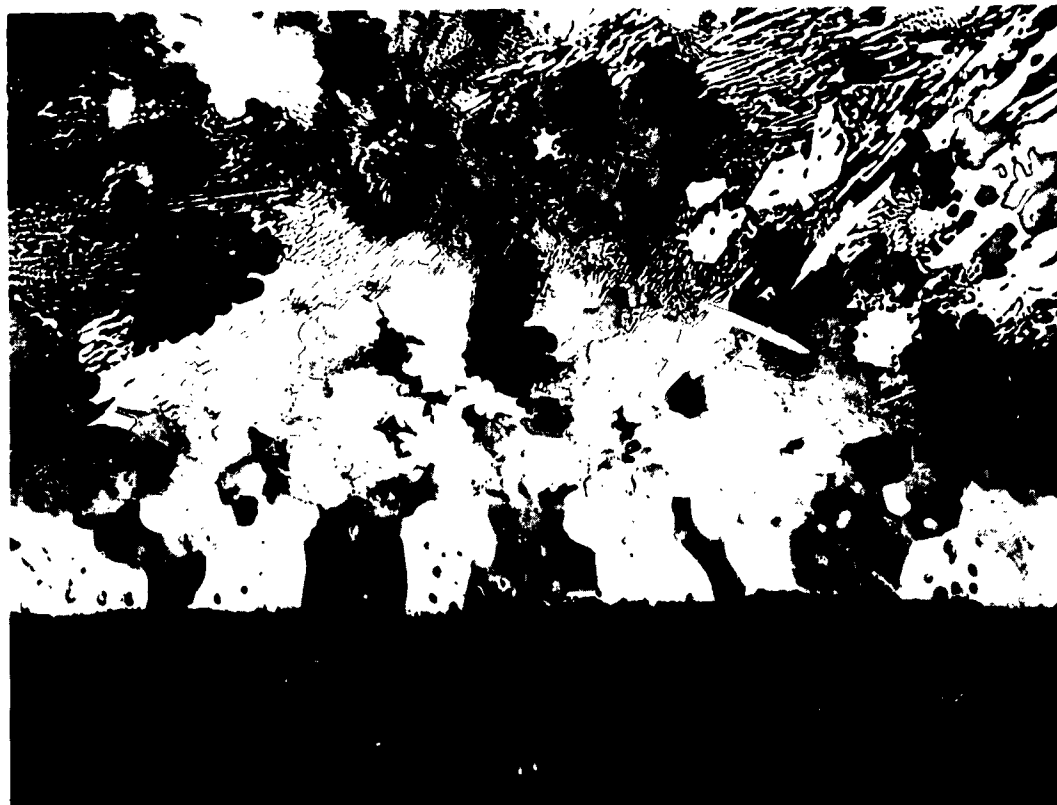


Figure 8 - Pre- and Post Aging Characteristics of Bare Copper Wire at Temperatures to 850°C in Purified Argon.



Brazing  
Alloy

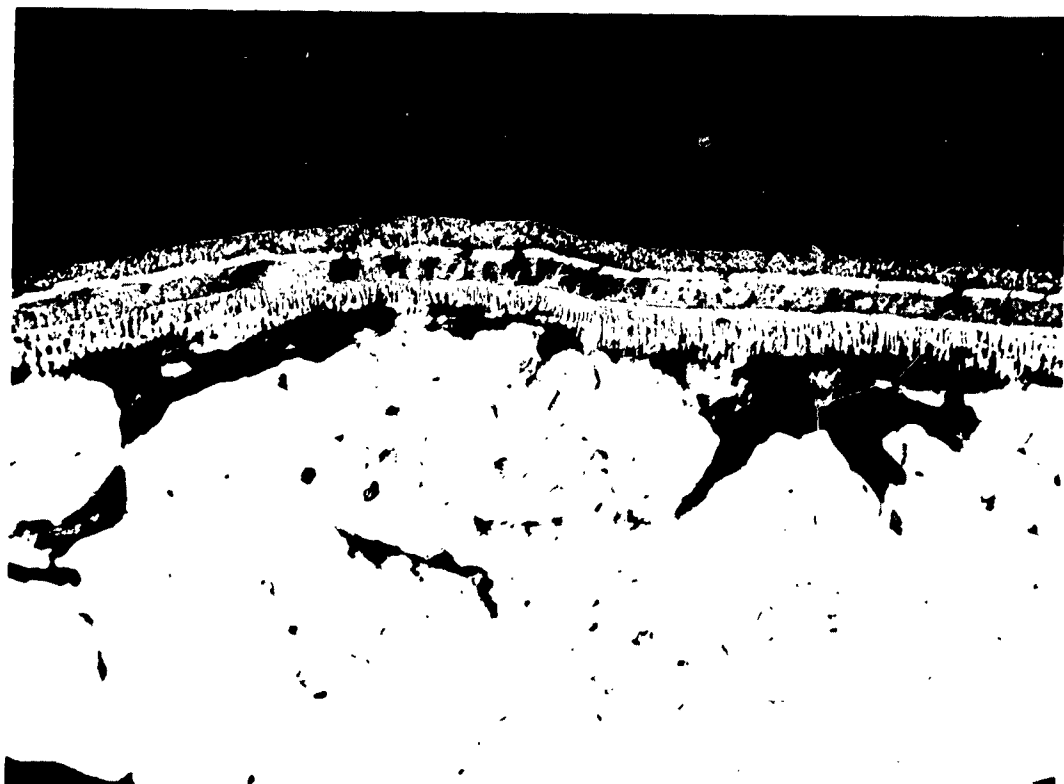
Interface

Lucalox

44845 250X LUCALOX/95ZR-.5BE C-1528

Figure 9 - Micrograph (250X) of Interface  
Between Lucalox and 95% Zr-5% Be  
Brazing Alloy.

Columnar grains at interface may indicate solution of  
Lucalox in brazing alloy. Joints have adequate strength but only  
poor to fair resistance to potassium at 850°C.



Lucaloc

Interface

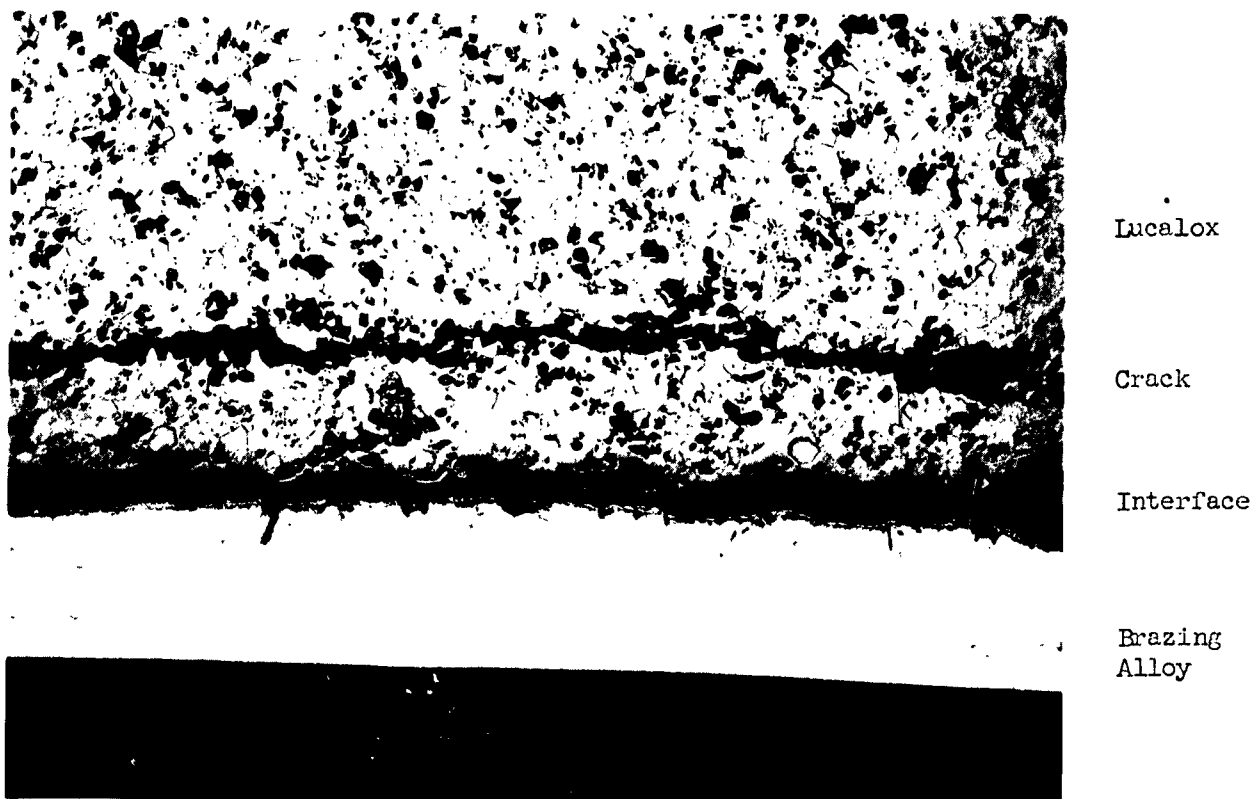
Brazing  
Alloy

45176 100X 67ZR-29V-4FE BRAZE ON  $Al_2O_3$  26D 01986

Figure 10 - Micrograph (100X) of Brazing Alloy Interface  
Exhibiting Bonded Structure.

The brazing alloy (67% Zr-29% V-4% Fe) exhibits a bonded structure, which probably consists of a solution of  $Al_2O_3$  in the brazing alloy. White light and unetched.





45186 100X 48ZR-48TI-4BE BRAZE TO  $AL_2O_3$  9D C1981

Figure 11 - Micrograph (100X) of Lucalox Specimen  
Showing Crack Formed when Brazing Alloy  
Cooled.

This specimen was wetted with 48% Beryllium-48% Titanium-4% Beryllium. Then cracking behavior was observed with several alloys, and the cracks are probably caused by solidification shrinkage and difference in the thermal coefficient of expansion.

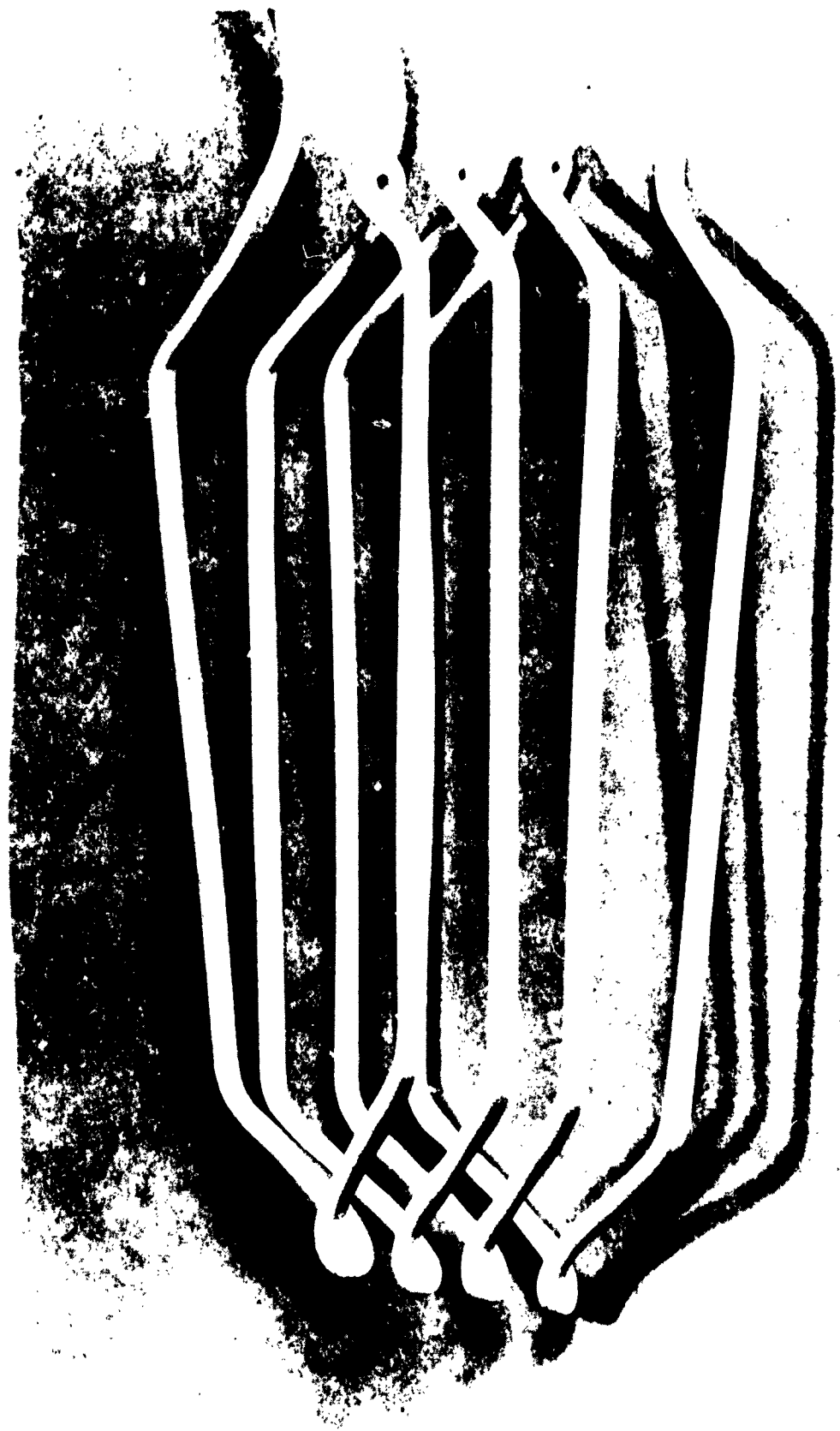


Figure 12 - Plasma sprayed alumina on pre-formed coil of nickel clad copper wire.



Figure 13 - Heavy pyrolytic aluminum nitride  
coating chipped from graphite rod.

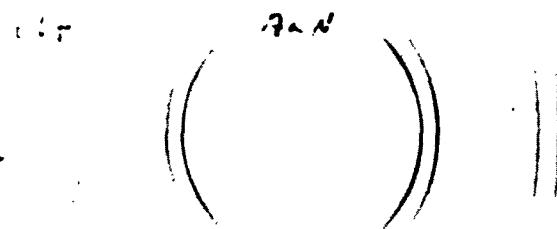


Figure 14 - X-ray Diffraction Pattern of  
Pyrolytic Aluminum Nitride Coating.

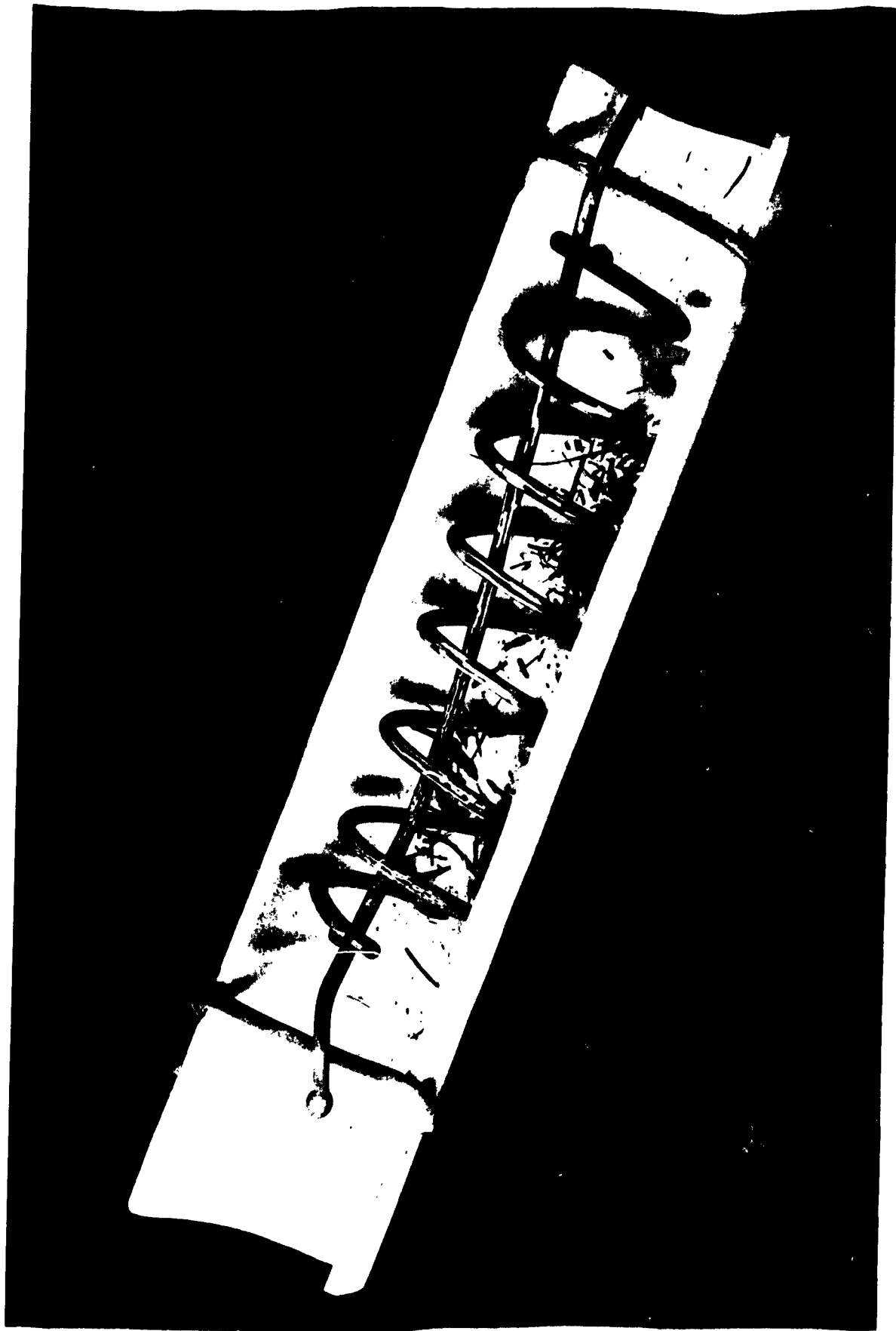


Figure 15 - Pyrolytic aluminum nitride on  
Oxalloy 28 wire.

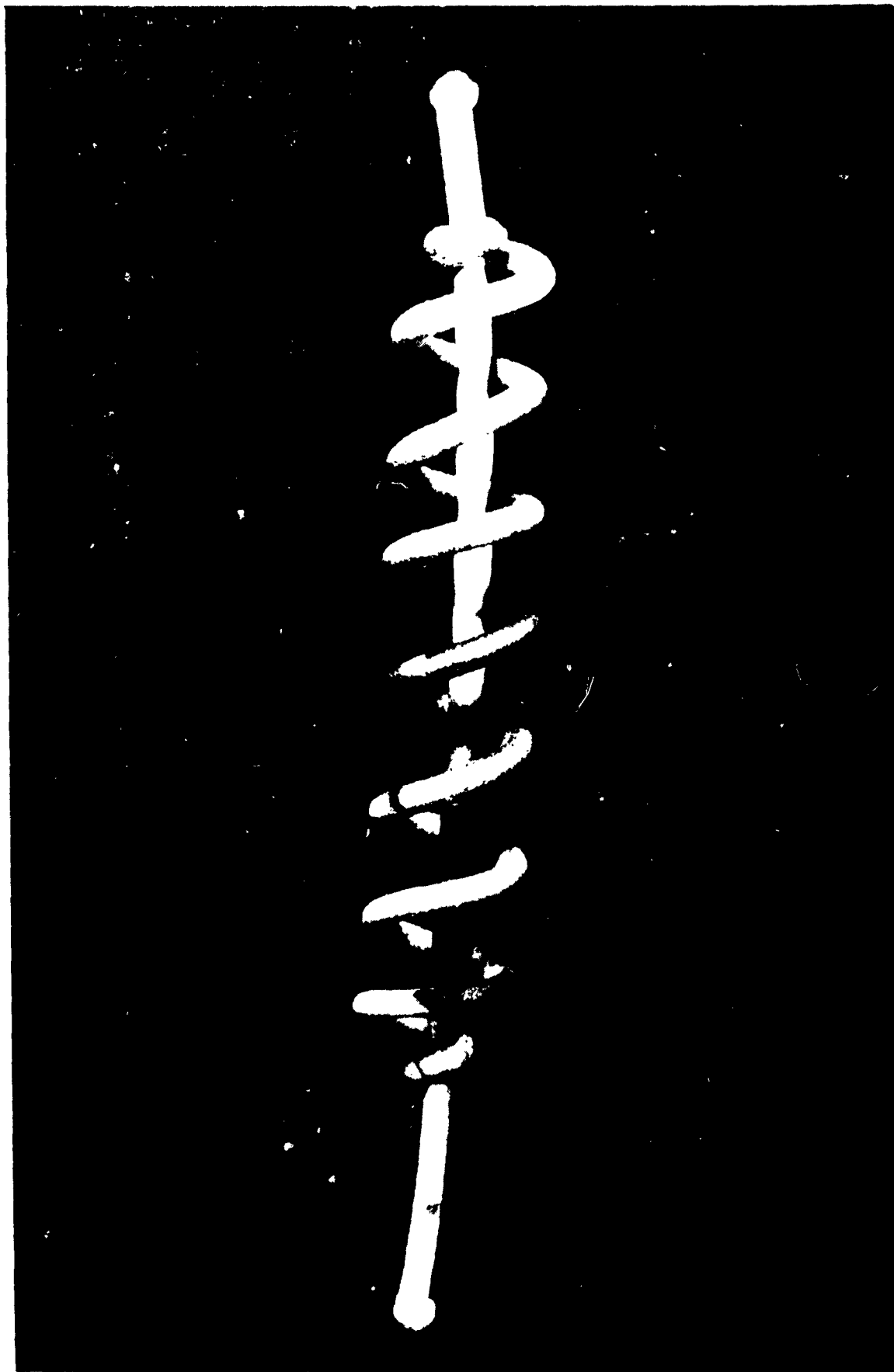


Figure 16 - Plasma sprayed alumina coating on Oxalloy 28 wire; after exposure to  $\text{AlCl}_3 \cdot \text{NH}_3$ . Wire temperature during exposure was  $875^\circ\text{C}$ .

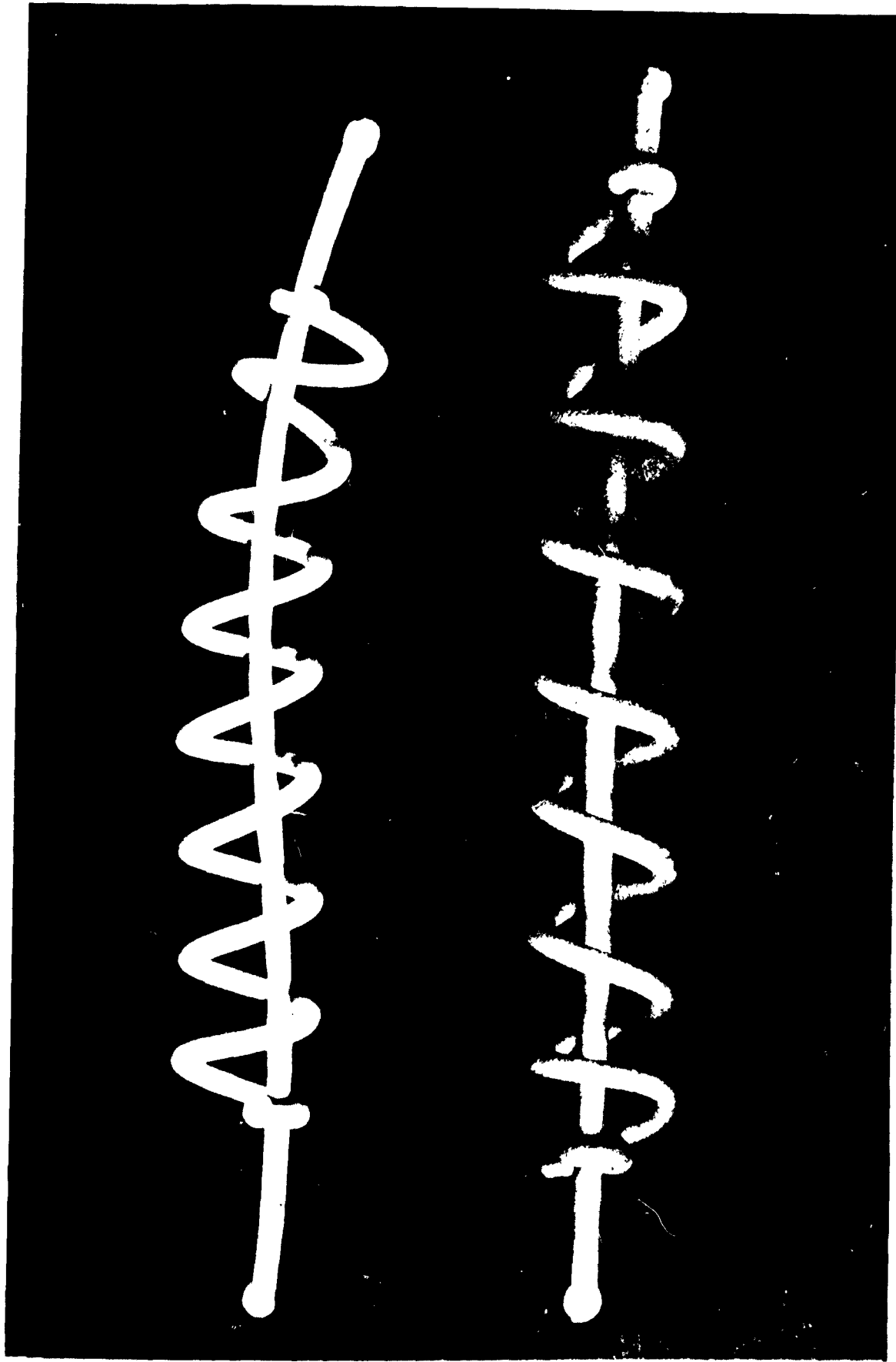
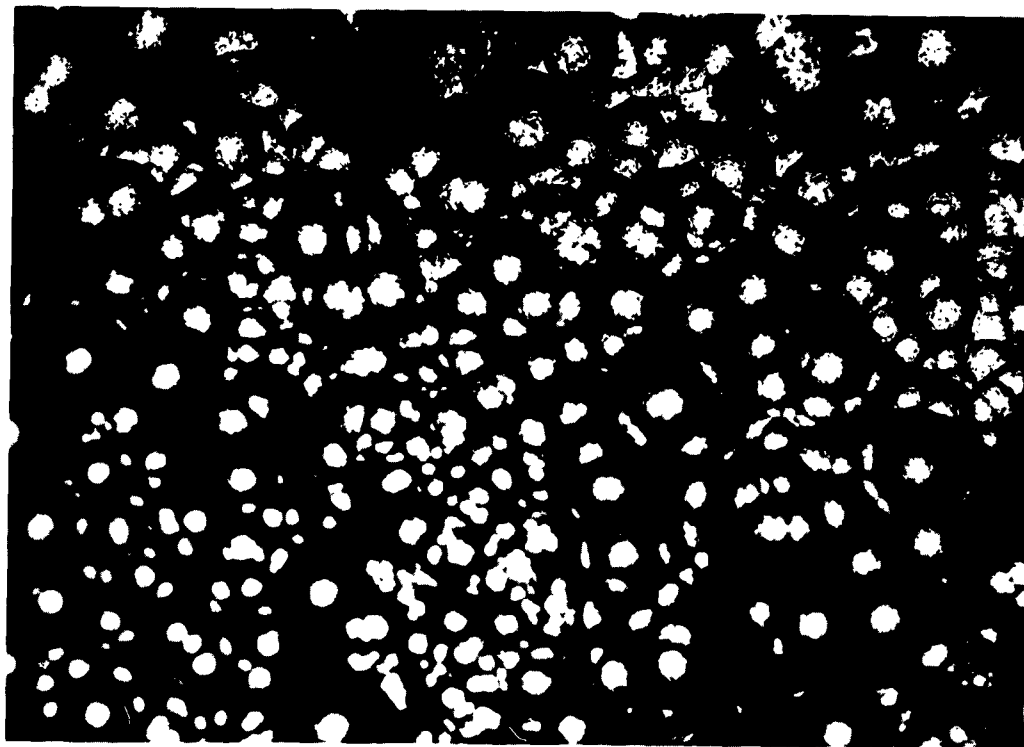


Figure 17 - Coated Oxalloy 28 coils.  
Top - Plasma sprayed alumina coated Oxalloy  
28 wire.  
Bottom - Similar alumina coated coil with  
pyrolytic aluminum nitride film.



Figure 18 - Carbon rods from BN forming work.  
Top - Untreated graphite rod.  
Center - Rod with BN film.  
Bottom - Rod with BN film exposed to K vapor.





44919 200X BN ON GRAPHITE ROD

Figure 19 - Photomicrograph (200X) of BN Coating  
Formed on a Graphite Rod.

Neg. 44919



Figure - Photomicrograph (200X) of EN Coating  
20 on Graphite (sample RB24-1) after 172 Hours  
Exposure to K Vapor at 850°C.

Neg. 45178

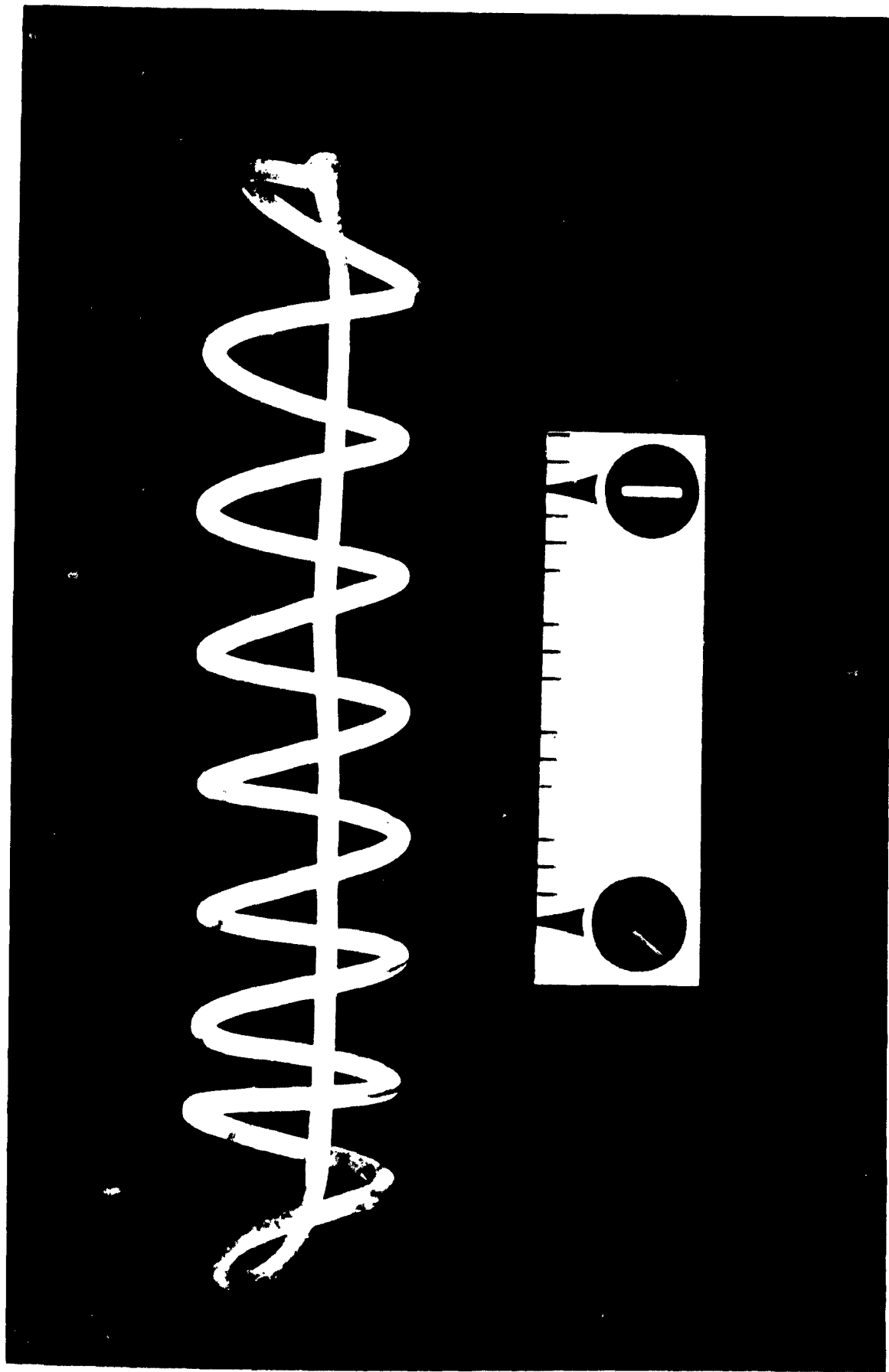


Figure 21 - BN coated Oxalloy 28 wire after  
exposure to K vapors (RB37-1)

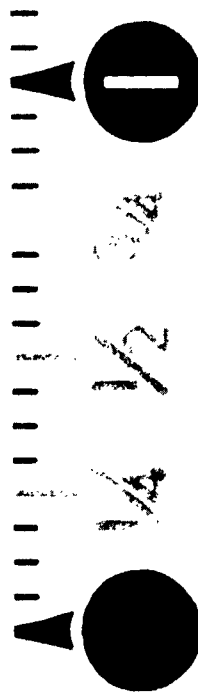
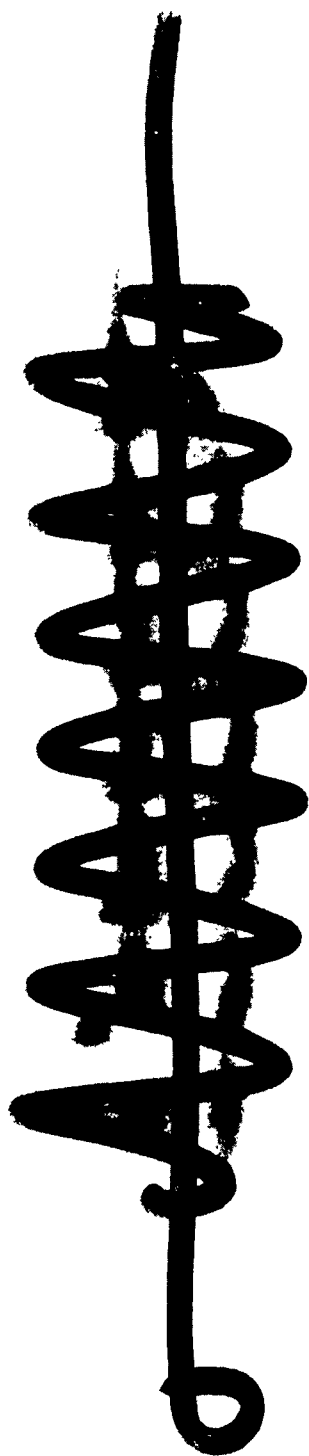


Figure 22 - BN coated Oxalloy 28 wire - BN film is transparent.

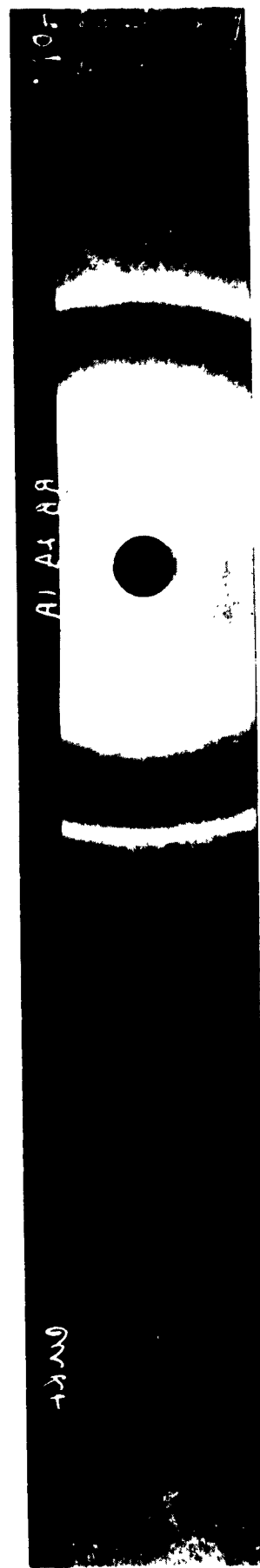
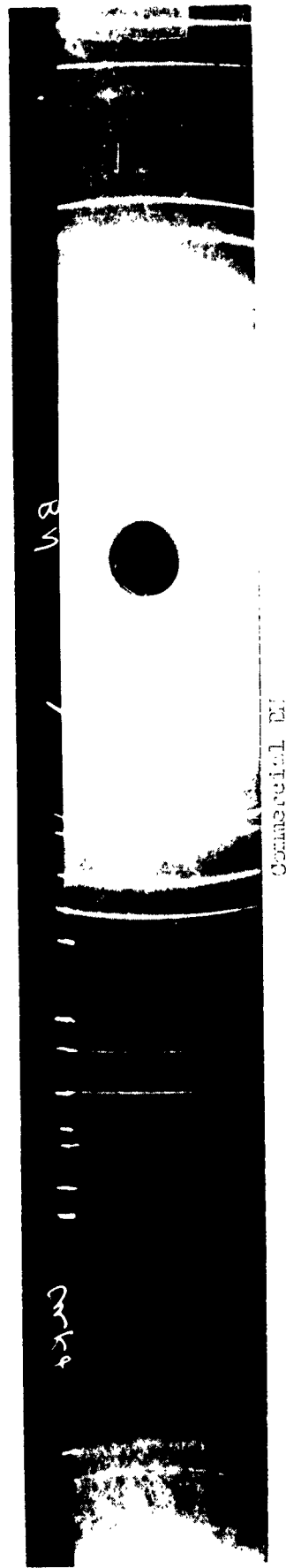
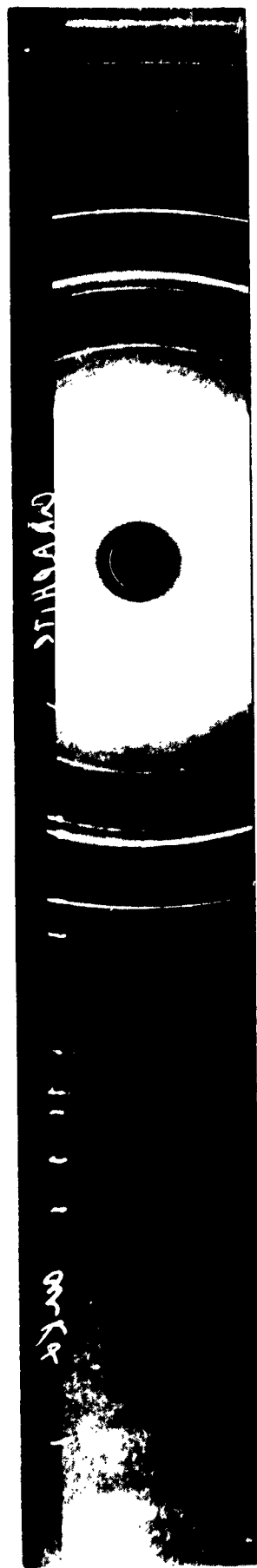


Figure 23 - X-ray Diffraction Patterns for Deposited Films

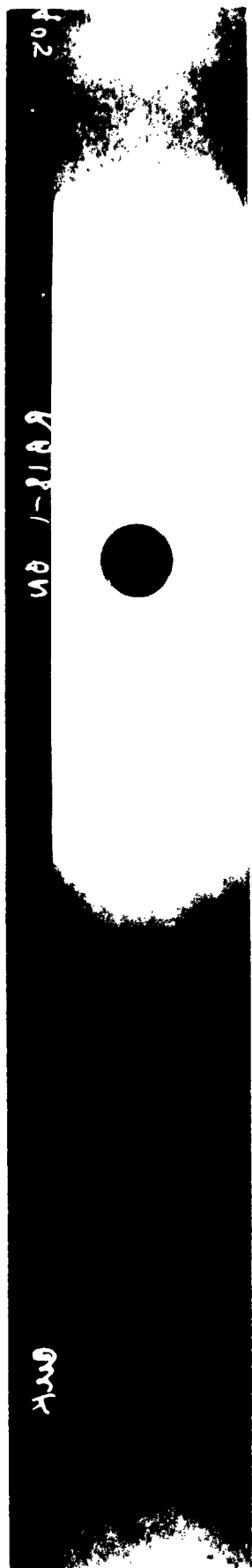
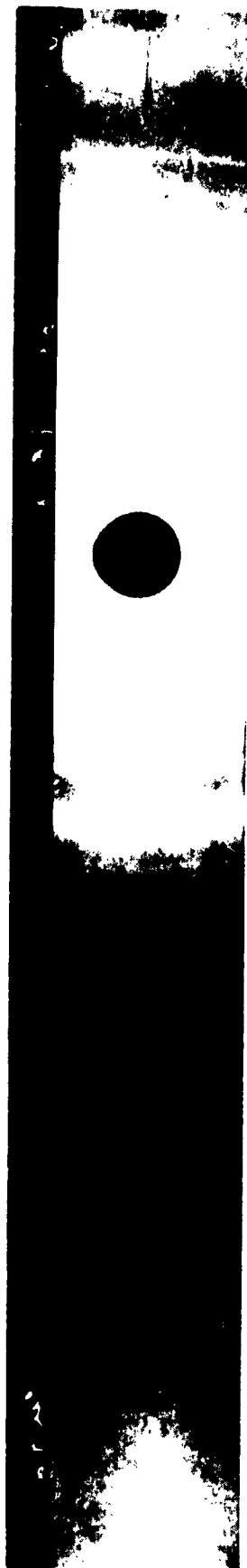


Figure 24 - X-ray Diffraction Patterns for Deposited Films



R 53 - 2

Figure 25 - X-ray Diffraction Patterns for Deposited Films



RB 27 - 1

Figure 27 - X-ray Diffraction Patterns for Deposited Films



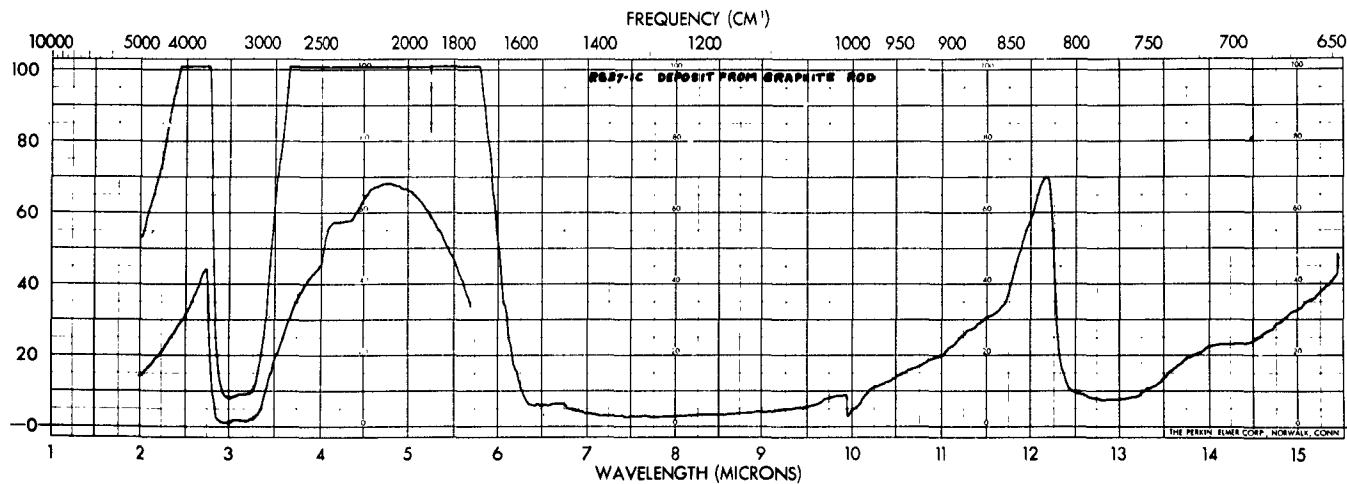
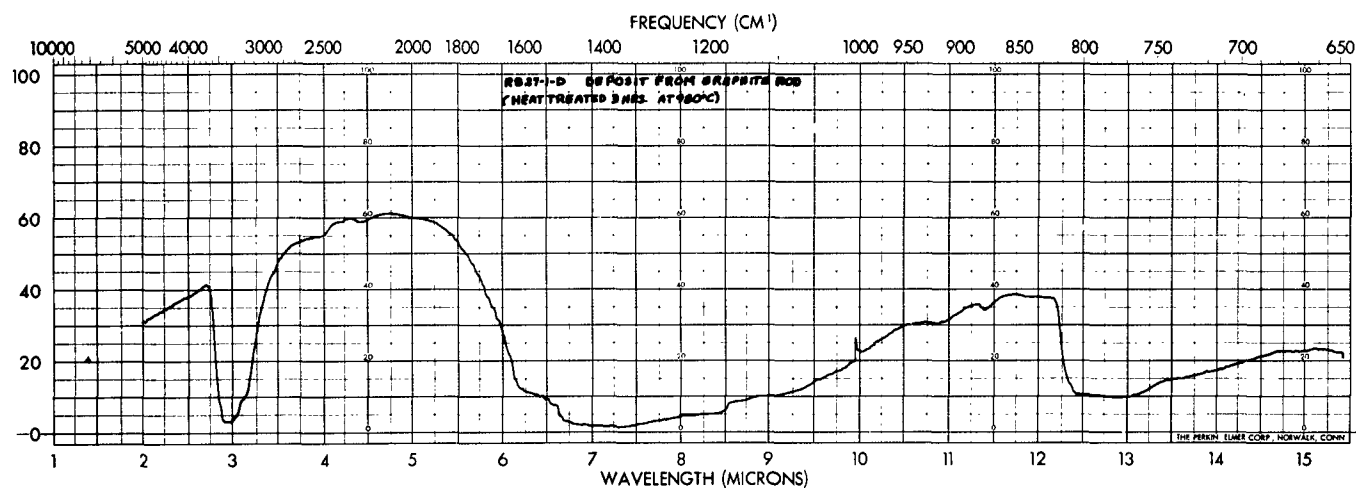
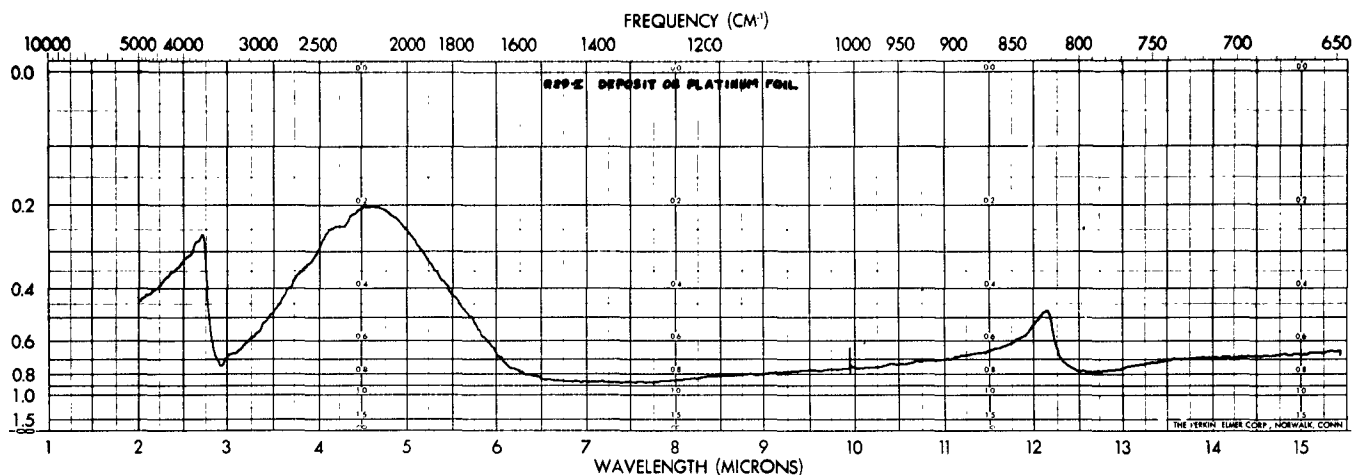


Figure 28 - Infrared spectra for specimens  
R29-I, RB27-1D and RB27-1C.

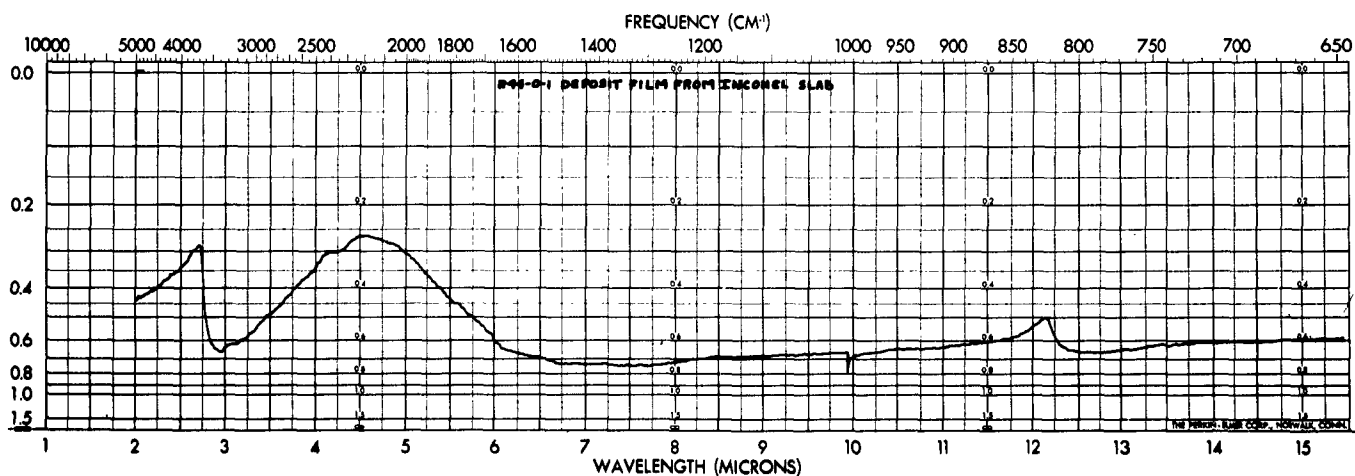
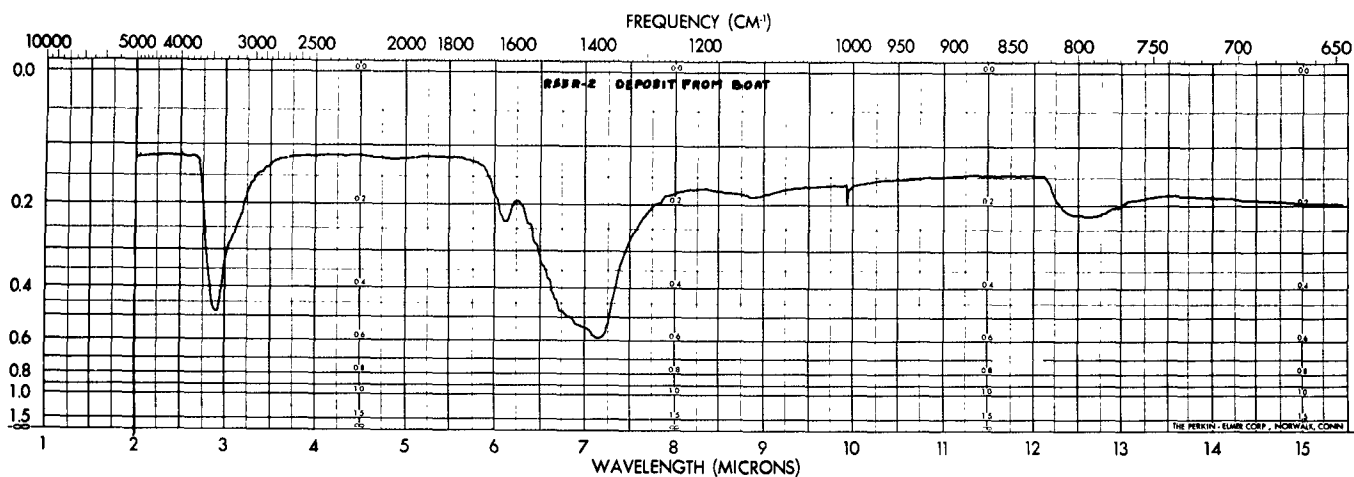
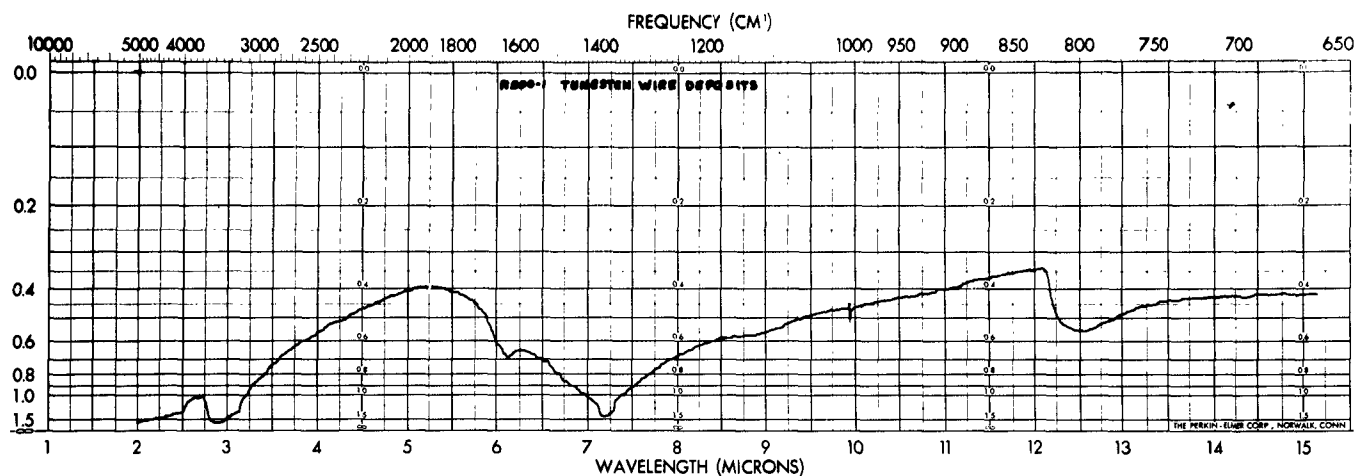


Figure 29 - Infrared spectra for specimens  
R50Q-1, R53R-2 and R44-O-1.

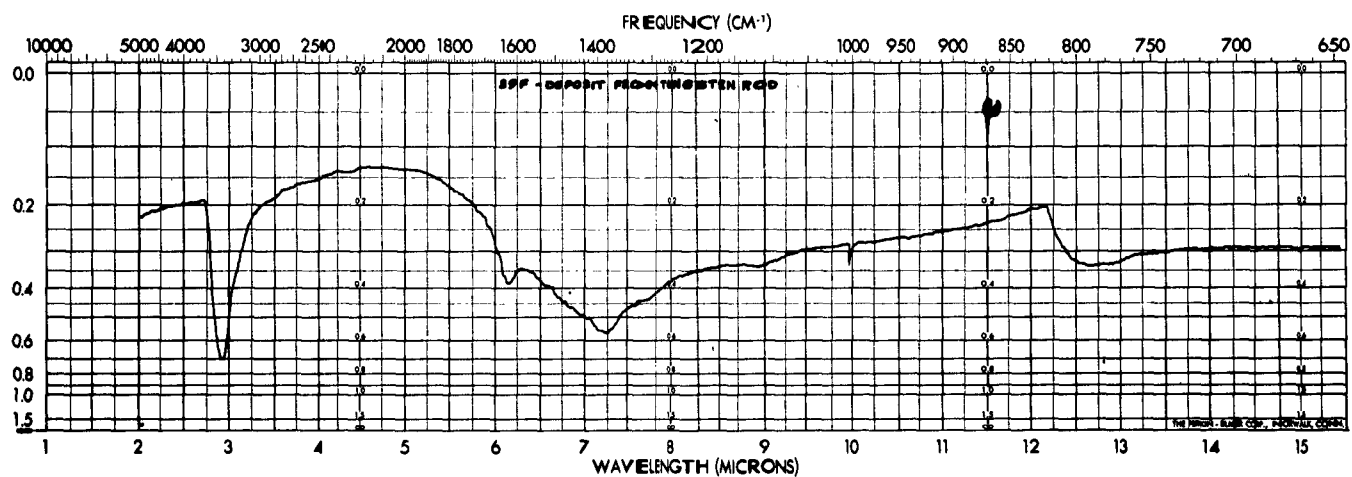
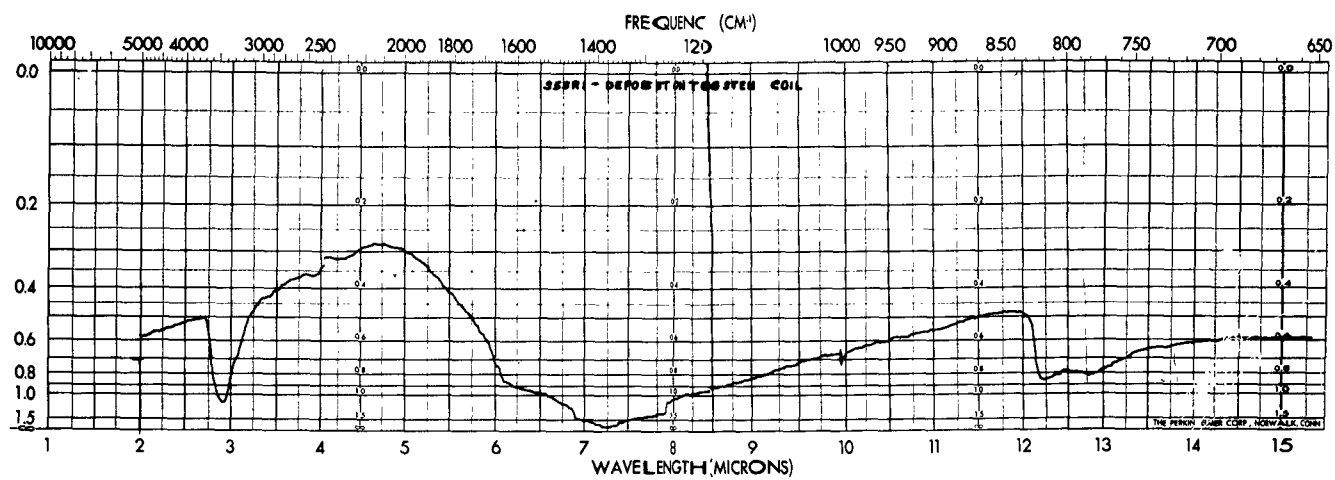
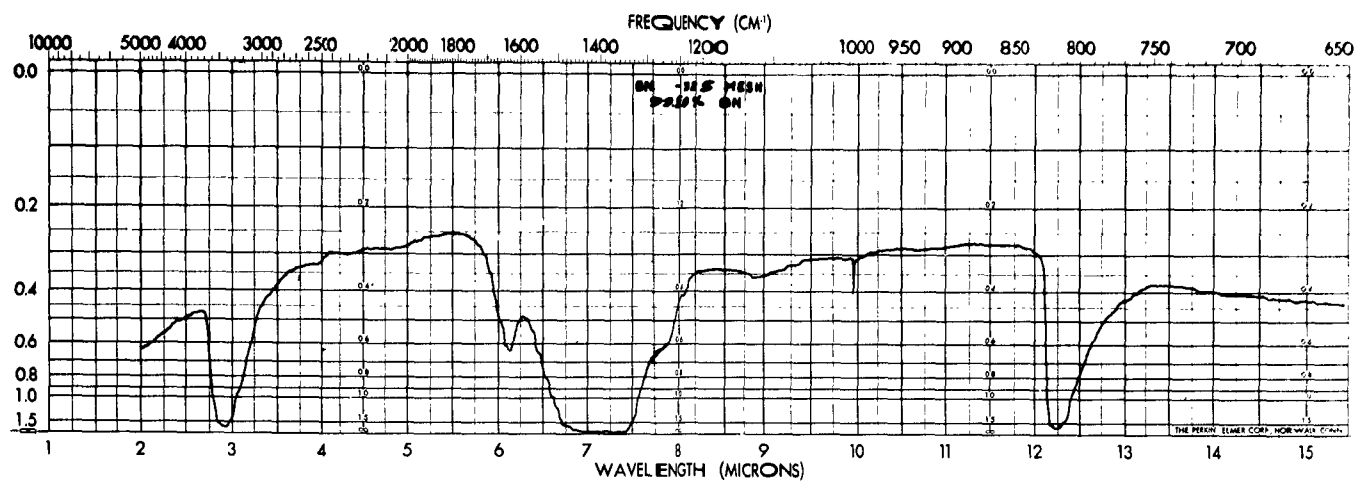


Figure 30 - Infrared spectra for specimens  
S53-R1, S9F and 99.5% BN.

TABLE 1

## POTASSIUM VAPOR EXPOSURES AT 850°C - 170 HOURS

Identity	Sample	Wt. Infor. - Grams		Comments
		Before	After	
CK - 14B	Beryllia	2.1912	2.1996	Light purple - hairline crack
CK - 14C	Beryllia	1.4271	--	Sample lost due to heavy capsule corrosion
CK - 17B	Boron Nitride-Sintered	.5504	--	Sample lost due to heavy capsule corrosion
CK - 18	Y-628 Strontium Zirconate	2.4631	2.4641	Light purple
CK - 19	Y-760 Strontium Zirconate	4.7779	4.7673	Mottled grey - Surface pocked
CK - 20	Al <sub>2</sub> O <sub>3</sub> -Flame Sprayed	2.0797	--	Al <sub>2</sub> O <sub>3</sub> lifted from metal surface
CK - 21	Sapphire Disk	.5423	--	Sample lost due to capsule corrosion
CK - 22	Ni Coated BeO	.1698	--	Sample lost due to capsule corrosion
CK - 23	Bn Coated Graphite	9.2922	--	Graphite cracked into pieces
CK - 24	Bn Film - 1000C Anneal	---	--	Film intact
CK - 25	Bn Film - 1000C Anneal	---	--	Film intact
CK - 26	Bn Film - No Anneal	---	--	Film intact
CK - 27	RC-38-2-AlN/Graphite	.0779	--	Sample still on test
CK - 28	RC-35-1-AlN/Graphite	2.4578	--	Sample still on test
CK - 29	RD7-1 Al <sub>2</sub> O <sub>3</sub> /AlN	4.1414	--	Sample still on test
SK - 2	Tube-to-Tube Ceramic Seal	10.0242	--	Sample lost due to heavy capsule corrosion
SK - 3	Ceramaseal	6.3993	--	Sample disintegrated
SK - 4	Stainless/Lucalox (95 Zr-5 Be)	.9985	--	Sample lost due to heavy capsule corrosion
SK - 5	Kovar/Sapphire (95 Zr-5 Be)	---	--	Sample lost due to heavy capsule corrosion
SK - 6	Kovar/Lucalox (95 Zr-5 Be)	.5433	.5481	Seal came apart
SK - 7	St. Steel/Lucalox (95 Zr-5 Be)	.6796	.6801	Seal came apart
SK - 8	St. Steel/Lucalox (95 Zr-5 Be)	.6561	.6568	Seal intact
SK - 9	Kovar/Lucalox (95 Zr-5 Be)	.5380	.5394	Seal intact
SK - 10	Ta/Lucalox (95 Zr-5 Be)	.9558	.9542	Seal came apart
SK - 11	Ta/Lucalox (95 Zr-5 Be)	.8566	.8572	Seal came apart

TABLE 1 (Continued)

Identity	Sample	Wt. Infor. - Grams		Comments
		Before	After	
WK - 8	Anodized Ta Rod	35.3413	--	Sample lost due to heavy capsule corrosion
WK - 9	Anodized Nb Rod	13.4054	--	Sample lost due to heavy capsule corrosion
WK - 10	Bn Coated Oxalloy	3.9993	--	Sample unchanged
BK - 1	45 Ti-40 Zr-15 Fe	.1191	.1205	Sample darkened
BK - 2	80 Zr-17 Fe-3 Be	.1747	.1812	Sample darkened-rough surface
BK - 3	48 Zr-48 Ti-4 Be (Ta/Lucalox)	.7953	.7884	Sample broke apart
BK - 4	75 Zr-19 Cb-6 Be (Ta/Lucalox)	.7408	.7408	Sample broke apart
BK - 5	62 Ti-26 Fe-8 Mo-4 Zr (Ta/Lucalox)	.6447	.5110	Sample broke apart
BK - 6	63 Ti-27 Fe-10 V (Ta/Lucalox)	.7963	.6974	Sample intact - capsule leak
BK - 7	63 Ti-27 Fe-10 Mo (Ta/Lucalox)	.5806	.5784	Sample intact
BK - 8	68 Ti-28 V-4 Be (Ta/Lucalox)	.5615	.5629	Sample intact
BK - 9	Microbrazed 130-Vacuum Heated	1.1684	--	Sample on test
BK - 10	Microbrazed 130-Argon Heated	.9790	--	Sample on test
BK - 11	28 Ni-72 Ti	.6326	--	Sample on test
BK - 12	62.5 Ti-37.5 Ni	.4429	--	Sample on test
BK - 13	Lucalox/Tantalum Joint	.3299	--	Sample on test
PK - 1	S7-63-1	2.0084	--	Disintegrated
PK - 2	S-30	1.8062	--	Disintegrated
PK - 3	VG-107	1.5521	--	Disintegrated
PK - 4	S-9	2.0909	--	Disintegrated
PK - 5	W-839	2.9806	--	Disintegrated
PK - 6	Alumina-101	14.1272	14.0544	Film badly attacked
PK - 7	ZrO-Hafnium Free XP-115	14.2052	14.2121	Sample film intact - purple
PK - 8	ZrO-201	14.4333	14.2469	Sample film intact - purple
PK - 9	15-A-7-SS	2.5685	2.5097	Heavy capsule corrosion
PK - 10	Alumina-105	14.3829	14.3878	Film lifted
PK - 11	Silica Free Al <sub>2</sub> O <sub>3</sub>	.8248	.8267	Turned gray, white powder leached out

TABLE 2

## MERCURY VAPOR EXPOSURE TESTS - 540°C

Identity	Sample	(Hrs) Time	Wt. Infor. - Grams			Comments
			Before	After	Change	
CH - 7B	Al <sub>2</sub> O <sub>3</sub> -Coran	260	2.0227	2.0146	-.0081	No apparent change
CH - 14B	Beryllia	260	2.3279	2.3277	.0002	No apparent change
CH - 18	Y-628 Strontium Zirconate	260	3.8928	3.8263	-.0065	No apparent change - Slight chip
CH - 19	Y-760 Strontium Zirconate	260	4.3932	4.3527	-.0405	Surface pocked
CH - 20	Al <sub>2</sub> O <sub>3</sub> -Flame Sprayed	260	2.0922	2.0918	-.0004	Surface darkened
CH - 22	Ni Coated BeO	260	.1545	.1530	-.0015	Surface black
WH - 5	Oxalloy	340	1.5008	1.4780	-.0228	Surface black
WH - 6	Cr Plated Cu-Both Ends Welded	340	1.0795	---	--	Cu core heavily attacked
WH - 6	Cr Plated Cu-One End Plated - One Weld	340	3.0935	---	--	Sample disintegrated
WH - 6	Cr Plated Cu-Both Ends Welded	340	3.1539	---	--	Sample disintegrated
WH - 7	Fe Plated Cu	340	.9936	---	--	Sample disintegrated
WH - 8	Anodized Ta	340	35.6448	35.6462	+0.02	Surface conductive after test
WH - 9	Anodized Nb	340	13.3753	13.3759	+0.0006	Surface conductive after test
SH - 2	Ceramic-to-Metal Seal	260	12.5337	---	--	Brazing alloy heavily attacked
SH - 3	Ceramaseal	260	6.7223	6.4121	-.3102	Brazing alloy attacked
SH - 5	Ceramaseal/SS Tube - Series 801		- - -	- - -	- - -	For electrical tests - - -
SH - 6	Ceramaseal/SS Tube - Series 805		- - -	- - -	- - -	For electrical tests - - -
SH - 7	Ceramaseal/SS Tube - Series 801		- - -	- - -	- - -	For electrical tests - - -
BH - 6	63 Ti-27 Fe-10 V	100	.5452	---	--	Sample now on test
BH - 8	68 Ti-28 V-4 Be	100	.1118	---	--	Sample now on test
BH - 12	62.5 Ti-37.5 Ni	100	.1303	---	--	Sample now on test
BH - 13	62.5 Ti-37.5 Ni (Lucalox/Ta Joint)	100	.8486	---	--	Sample now on test
BH - 14	95 Zr-5 Be	100	.0846	---	--	Sample now on test
BH - 15	71.5 Ti-28.5 Ni	100	.1327	---	--	Sample now on test

TABLE 3

AVERAGE MEASURED ELECTRICAL CONDUCTIVITY OF VARIOUS CONDUCTOR SAMPLES  
WHILE THERMAL AGING AT 538°C FOR 500 HOURS IN ARGON

Sample	Samples "As Received"		Samples Pre-exposed to Mercury Vapors	
	Conductivity, mhos-cm <sup>-1</sup>	% Calculated Conductivity of Copper at 538°C	Conductivity, mhos-cm <sup>-1</sup>	% Calculated Conductivity of Copper at 538°C
Bare Commercial Copper	1.82 x 10 <sup>5</sup>	102	---	---
Chrome-Plated Copper	1.73 x 10 <sup>5</sup>	97	---	---
Iron-Plated Copper	1.55 x 10 <sup>5</sup>	87	---	---
"Oxalloy 28"***	1.37 x 10 <sup>5</sup>	77	*1.35 x 10 <sup>5</sup>	77

\* Pre-exposed to 538°C mercury vapor for 360 hours.

\*\* Stainless steel clad-copper conductor (28% S/S by vol.).

TABLE 4

AVERAGE MEASURED CONDUCTIVITY OF VARIOUS CONDUCTOR SAMPLES  
WHILE THERMAL AGING AT 850°C FOR 500 HOURS IN ARGON

Sample	Samples "As Received"	
	Conductivity, mhos-cm <sup>-1</sup>	% Calculated Conductivity of Copper at 850°C
Bare Commercial Copper**	1.35 x 10 <sup>5</sup>	103
Nickel Clad - Silver*	1.1 x 10 <sup>5</sup>	89
"Oxalloy 28"	Failed after 400 hours***	

\* 20% Nickel Clad - Silver

\*\* 28% Stainless Steel Clad - Copper

\*\*\* Test of a second specimen failed after 100 hours



TABLE 5

ELECTRICAL VOLUME RESISTIVITY OF VARIOUS MATERIALS  
AT 538°C IN ARGON

<u>Material</u>	<u>Volume Resistivity, Ohm-Inches</u>	
	<u>"As Received"</u>	<u>"Pre-exposed"*</u>
(CX-14) Beryllia	$1.7 \times 10^{10}$	---
(CH-14) Beryllia	---	$1.1 \times 10^4$
(CX-12) Sapphire	$6.5 \times 10^9$	---
(CX-17B) Sintered Boron Nitride	$6.0 \times 10^8$	---
(CX-10) Hot Pressed Magnesia	$1.9 \times 10^7$	---
(CH-7B) Alumina (Coran)	---	$3.5 \times 10^5$
(CH-18) Strontium Zirconate (Y-628)	---	$1.4 \times 10^7$
(CH-19) Strontium Zirconate (Y-760)	---	$4.2 \times 10^4$

\*Pre-exposed to 538°C mercury vapor for 260 hours.

TABLE 6

ELECTRICAL VOLUME RESISTIVITY OF VARIOUS MATERIALS  
AT 850°C IN ARGON

<u>Material</u>	<u>Volume Resistivity, Ohm-Inches</u>	
	<u>"As Received"</u>	<u>"Pre-exposed"*</u>
(CX-12) Sapphire	$1.5 \times 10^7$	---
(CX-14) Beryllia	$6.0 \times 10^6$	---
(CX-10) Hot Pressed Magnesia	$2.5 \times 10^6$	---
(CK-10) Hot Pressed Magnesia	---	$7.8 \times 10^3$
(CX-17B) Sintered Boron Nitride	$6.0 \times 10^5$	---
(CK-19) Strontium Zirconate (Y-760)	---	$1.4 \times 10^4$
(CK-18) Strontium Zirconate (Y-628)	---	$6.5 \times 10^4$

\*Pre-exposed to 850°C potassium vapor for 165 hours.

TABLE 7

WETTING PROPERTIES AND RESISTANCE TO POTASSIUM  
OF SOME ACTIVE METALS ON PURE  $Al_2O_3$

Resistance									
To Potassium: 170 Hr Exposure at 850°C									
Quality of Wetting on Pure Al2O3									
Ti	Zr	Fe	Cb	Be	V	Mo	Point °C	Remarks	
--	67	4	--	--	29	--	1300°C	Not tested	---
--	60	--	15	--	25	--	1280°C	Not tested	---
48	48	--	--	4	--	--	1050°C	Alloy separated from alumina in test. Attack occurred at interface.	Alloy penetrates into and dissolves alumina during brazing.
63	--	--	--	--	27	10	1250°C	Alloy could be separated from alumina after exposure. Separation occurred at alumina-brazing alloy interface.	---
63	--	27	--	--	10	--	1280°C	Test capsule leaked. Test to be repeated.	---
68	--	--	--	4	28	--	1250°C	Joint intact after exposure but could be removed with some difficulty. Fracture occurred in crack already present before exposure.	This alloy shows some promise, but Lucalox disk cracked when joined to tantalum tube with this alloy.
45	40	15	--	--	--	--	1050°C	Not tested.	---
--	75	--	19	6	--	--	1050°C	Brazing alloy and tantalum separated from Lucalox in test.	Some columnar grains at interface.
46	46	--	--	4	4	--	1000°C	Not tested.	---
62	4	26	--	--	--	8	1200°C	Alloy separated from Al2O3 at interface.	---

TABLE 7 (Continued)

<u>Ti</u>	<u>Zr</u>	<u>Fe</u>	<u>Cb</u>	<u>Be</u>	<u>V</u>	<u>Mo</u>	<u>Point °C</u>	Quality of Wetting On Pure Al <sub>2</sub> O <sub>3</sub>	Resistance To Potassium: 170 Hr Exposure at 850°C	Remarks
--	95	--	--	5	--	--	980°C	Excellent wetting.	Alloy separated from Al <sub>2</sub> O <sub>3</sub> at interface.	Several leak tight Lucalox disk to tanta- lum tube joints have been made. These are to be tested in potas- sium and mercury.

TABLE 8

PLASMA-SPRAYED MATERIALS AND SUBSTRATES USED

<u>Oxides</u>	<u>Substrates</u>
Modified Alumina (Metco 101)	Inconel Strip Nickel Rod
Pure Alumina (Metco 105)	Inconel Strip Inconel Clad Copper Stainless Steel Clad Copper Nickel Bar
Zirconia, Hafnium Free Lime Stabilized (Metco XP-1115)	Inconel Strip Nickel Bar
Zirconia (Metco 201)	Inconel Strip
Magnesia (Norton)	Inconel Strip Nickel Bar
<u>Non-Oxides</u>	
Nickel-Aluminide (Metco 404)	Inconel Strip Nickel Bar

TABLE 9

SAMPLE SUBSTRATES AND TEMPERATURES  
USED TO FORM BN COATINGS

<u>Sample</u>	<u>Substrate</u>	<u>Substrate Temperature (°C)</u>
RB241A	Graphite	1445
RB18-1	Platinum	1000
R500-1	Stainless Steel Clad Cu	925
RB24-1	Graphite	1445
R4401	Inconel	1155
RB24-1B	Graphite	Annealed at 980°C for 3 hrs. in air
R53R-2	(Boat Deposit)	
RB11X-1	Stainless Steel Rod	1005
R53R-1	Tungsten Wire	1950
RB27-1	Graphite	1375
R29I-1	Platinum Cylinder	1300
S13M	Platinum Wire Coil	1375

TABLE 10

X-RAY DATA FOR FILM DEPOSITS\*

Sample	d Values from X-ray Data									
	3.31	2.16	2.05	1.81	1.66	1.25	1.17	1.00		
BN (ASTM)	3.31	2.16	2.05	1.81	1.66	1.25	1.17	1.00		
Graphite (ASTM)	3.35	2.12	2.03	--	1.67	1.225	1.15	.98		
RB241A	3.40B**	2.12B	--	--	1.70B	1.23B	--	--		
RB18-1	3.40B	2.12B	--	--	--	1.24B	1.16B	--		
R50Q-1	3.45B	2.14B	--	--	1.72B	1.24B	--	--		
RB24-1	3.50B	2.12B	--	--	1.70B	1.23B	--	--		
R440-1	3.40B	2.11B	--	--	1.72B	1.23B	--	--		
RB241B	3.40B	2.10B	--	--	--	1.22B	--	--		
R53R-2	--	--	--	--	AMORPHOUS	--	--	--		
RB11X-1	3.40B	2.11B	--	--	--	1.22B	--	plus Fe <sub>2</sub> O <sub>3</sub> and Fe <sub>3</sub> O <sub>4</sub>		
R53R-1	3.40B	2.12B	--	--	1.72B	1.23B	1.17B			
RB27-1	3.40B	2.10B	--	--	--	1.23B	--	--		
R29I-1	3.25B	2.10B	--	--	1.70B	1.23B	--	--		

\*All patterns taken in 114-6 mm Debye Scherrer powder cameras using Nickel filtered copper radiation.

\*\*Designation "B" denotes broad line.